Historic, archived document

Do not assume content reflects current scientific knowledge, policies, or practices.

42B
REYNOLDS LI
LIBRARY
RECEIVED
JUL 25 1949
U. S. DEPT. OF AGRICULTURE
C&R.S

U. S. DEPARTMENT OF AGRICULTURE.
DIVISION OF CHEMISTRY,
BULLETIN No. 35.

PROCEEDINGS

OF THE

NINTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

THE NATIONAL MUSEUM, WASHINGTON, D. C.,

AUGUST 25, 26, AND 27, 1892.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.

PUBLISHED BY AUTHORITY OF THE SECRETARY OF AGRICULTURE.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1892.

U. S. DEPARTMENT OF AGRICULTURE.
DIVISION OF CHEMISTRY.
BULLETIN No. 35.

PROCEEDINGS

OF THE

NINTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

THE NATIONAL MUSEUM, WASHINGTON, D. C.,

AUGUST 25, 26, AND 27, 1892.

EDITED BY
HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.

PUBLISHED BY AUTHORITY OF THE SECRETARY OF AGRICULTURE.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1892.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., September 24, 1892.

SIR: I beg to submit herewith, for your inspection and approval, the manuscript of the proceedings of the ninth annual convention of the Association of Official Agricultural Chemists.

Respectfully,

H. W. WILEY,
Chemist.

Hon. J. M. RUSK,
Secretary of Agriculture.

CONTENTS.

FIRST DAY—MORNING SESSION.

Page.

List of members and visitors present 1
Order of business 2
Annual address of the president 2
Report on dairy products 7

FIRST DAY—AFTERNOON SESSION.

Report on phosphoric acid 23
Paper: Determinations of loss on heating samples of phosphates sent out by the reporter on phosphoric acid for 1892 47
Paper: The occurrence of metaphosphoric and pyrophosphoric acids in cotton-seed meal 50
Paper: The use of ammonium nitrate in the determination of phosphoric acid .. 53

SECOND DAY—MORNING SESSION.

Report on potash 58
Report on methods for the determination of nitrogen 67
Paper: Comparison of methods for the standardization of acid and alkaline solutions 80
Paper: The Gunning-Kjeldahl method and a modification applicable in the presence of nitrates 85
Paper: The Ulsch method in determining nitric nitrogen in complete fertilizers 88

SECOND DAY—EVENING SESSION.

Report on soils and ash 93
Address of Hon. Edwin Willits 98
Election of officers 100
Miscellaneous business 100

THIRD DAY—MORNING SESSION.

Report on cattle foods low in carbohydrates 111
Report on foods and feeding stuffs low in carbohydrates 127
Report on sugar 137
Report on fermented liquors 171
Chemists connected with the State agricultural experiment stations 186
Chemists connected with agricultural colleges 188
Chemists connected with the U. S. Department of Agriculture 190
Chemists connected with the U. S. Treasury Department 191

Page.
Officers, reporters, and committees of the Association of Official Agricultural Chemists of the United States for 1892–'93 192
Constitution of the Association of Official Agricultural Chemists 193
Official methods of analysis adopted by the Association of Official Agricultural Chemists, August 25–27, 1892 195
Methods of determining phosphoric acid 195
Methods of determining potash 197
Methods for the determination of nitrogen 198
Methods of sugar analysis 204
Methods of analysis of foods and feeding stuffs 214
Methods of analysis of dairy products 216
Methods of analysis for soils and ash 225
Methods of analysis of fermented liquors 232
Table of atomic weights 243

PROCEEDINGS OF THE NINTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, HELD AT WASHINGTON, AUGUST 25, 26, AND 27, 1892.

FIRST DAY.

MORNING SESSION, THURSDAY.

In accordance with the call of the executive committee, the association met in the lecture hall of the U. S. National Museum, at 10 o'clock, the president, Mr. N. T. Lupton, in the chair.

The following members, and others interested in the objects of the association, were present:

Hon. Edwin Willits, Assistant Secretary of Agriculture.

The president, Mr. N. T. Lupton, Auburn, Ala.

The vice-president, Mr. S. M. Babcock, Madison, Wis.

The secretary, Mr. H. W. Wiley, Chief Chemist, U. S. Department of Agriculture.

Of the executive committee, Messrs. Penny and Huston, and the following members and participants:

Allen, E. W., Washington, D. C.
Anderson, J. T., Auburn, Ala.
Armsby, H. P., State College, Pa.
Beal, W. H., Washington, D. C.
Black, S. E., Washington, D. C.
Carman, J. Y., Washington, D. C.
Cathcart, C. S., New Brunswick, N. J.
Chace, E., Washington, D. C.
Clarke, R. W., Washington, D. C.
Crampton, C. A., Washington, D. C.
Davidson, R. J., Blacksburg, Va.
De Roode, Rudolph J. J., Morgantown, W. Va.
De Schweinitz, E. A., Washington, D. C.
Dugan, John, Washington, D. C.
Ewell, E. E., Washington, D. C.
Fellows, G. S., Washington, D. C.
Frear, William, State College, Pa.
Hardee, R. E., Experiment, Ga.
Hardin, M. B., Fort Hill, S. C.
Harrington, H. H., College Station, Texas.
Harris, A. W., Washington, D. C.
Holton, Frederick A., Washington, D. C.
Huston, H. A., La Fayette, Ind.
Hutchinson, W. L., Agricultural College, Miss.
Hutchinson, P. L., Atlanta, Ga.
Kedzie, R. C., Agricultural College, Mich.
Kilgore, B. W., Raleigh, N. C.
Knorr, A. E., Washington, D. C.
Krug, William H., Washington, D. C.
Lehmann, A., Ottawa, Canada.
McDonnell, H. B., College Park, Md.
McElroy, K. P., Washington, D. C.
Parsons, C. L., Durham, N. H.
Patterson, H. J., College Park, Md.
Payne, George F., Atlanta, Ga.
Penny, C. L., Newark, Del.
Peter, A. M., Lexington, Ky.
Robinson, Norman, Tallahassee, Fla.
Ross, B. B., Baton Rouge, La.
Scovell, M. A., Lexington, Ky.
Shiver, F. S., Fort Hill, S. C.
Spencer, G. L., Washington, D. C.
Street, John P., New Brunswick, N. J.
Terne, Bruno, Philadelphia, Pa.
Van Slyke, L. L., Geneva, N. Y.
Von Herff, B., Washington, D. C.
Voorhées, E. B., New Brunswick, N. J.
Voorhees, L. A., New Brunswick, N. J.
Wedderburn, Aug., Washington, D. C.
Wheeler, H. J., Kingston, R. I.
Whitney, Milton, Baltimore, Md.
Winton, A. L., New Haven, Conn.
Woods, Charles D., Middletown, Conn.
Yeates, W. S., Washington, D. C.

The president having called the association to order, Mr. Wiley moved that the chair appoint a committee of three on the order of business. Carried.

The chair appointed as such committee Messrs. Wiley, Babcock, and Scovell.

Mr. Wiley presented the following report of the committee on order of business.

PROGRAMME.

HOURS OF MEETING.—Morning hours, 9:30 to 12:30; afternoon hours, 2 p. m., extending indefinitely to time of adjournment.

The order of business is as follows:

1. President's address.
2. Report on phosphoric acid.
3. Report on potash.
4. Report on nitrogen.
5. Report on soils and ash.
6. Report on dairy products.
7. Report on foods rich in carbohydrates.
8. Report on foods poor in carbohydrates.
9. Report on sugar.
10. Report on fermented liquors.

By special order the business meeting was set at 3 p. m., Friday, August 26.

On motion of Mr. Kedzie the report was accepted and adopted.

Immediately following the report of the committee on order of business the president gave his annual address:

ADDRESS OF MR. N. T. LUPTON, PRESIDENT.

GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: Like my predecessors in office, I have been somewhat perplexed in the selection of a subject or line of thought for an opening address. The time at my disposal is too short, if the occasion was opportune, for a thorough discussion of any of the important questions which concern the practical worker in the laboratory, while, for the details of methods, we have reporters who will, in due time, present the results of their study and investigation since our last meeting, assisted as they have been by the active members of the association.

This coöperation of trained chemists throughout our wide-extended country, under the direction of reporters, annually appointed to direct investigation and to collect, tabulate, and discuss results, is undoubtedly one of the most valuable features of our organization. I shall not anticipate these reports by discussing methods of analysis, but call attention briefly to some points connected with the responsible work committed to our hands which seem to deserve especial mention.

First, a word in reference to conformity to our prescribed methods. All agree that these should be strictly adhered to in our published results. As public analysts, our certificates are taken in evidence by

the courts of the country, and their money value is of very great importance to manufacturers and others, to say nothing of their higher value as reliable expressions of the composition of the great variety of substances submitted to us for analyses. If these methods are unsatisfactory to any member of the association, the amplest opportunity is given in our annual meetings for the presentation of objections and the adoption of changes, should such be desirable in the opinion of this body. Until such changes are officially made, it is the duty of each and every analyst to conform strictly to the instructions given in our bulletins. Should it, for good and sufficient reasons, be deemed advisable to deviate from official methods, let the results be so stated in unequivocal language. Those who have had much experience in such matters know the disposition of young chemists to make short cuts, and, in a spirit of perfect honesty, to make slight changes here and there in the methods prescribed for their guidance.

A close examination of the published work of the association will show, I think, a gratifying approximation to uniformity in results, and yet there are perplexing discrepancies not easily accounted for, otherwise than on the ground of deviation from the directions given or want of care in the work. These discrepancies are in some instances too great to be laid to "personal error," as in astronomical and other observations. Perfect uniformity, even in our individual work, is practically unattainable, but a close approximation is not only possible but eminently desirable.

In the language of a recent writer—

> The hand, of course, is not always steady, nor is the eye always accurate, and while we are liable to physical defects and weaknesses, we shall never be free from mistakes; but it is nevertheless a fact, which has forced itself upon all thinking men, that uniformity in manipulation is the prime factor in the attainment of uniform results, and nowhere is such uniformity a *sine qua non* as in the laboratory.

A recent communication from the representative in this country of the principal potash mines of Germany calls attention to an inclosed description and critique of various methods of potash determination, by the chief chemist of the Anhalt mines, in which the American official methods are unfavorably commented upon and pronounced to be less accurate and more time-consuming than the one used in the potash industry referred to. Of course chemists differ as to methods, and give up with reluctance those to which they have become attached by long usage, but we are not so wedded to ours as to hesitate to adopt changes when convinced of their wisdom. The Lindo-Gladding method has given pretty general satisfaction, and while differences are shown in the table of results reported to this association two years ago and reproduced in the communication referred to, these differences are by no means excessive, and by excluding two or three of the reports in which the results obtained may perhaps not be chargeable to the method used, there is a close agreement.

Let no one, however, infer that we object to criticism or to the closest scrutiny of our work, but in a spirit of true conservatism no changes

can be made unless convinced of their desirability after the most careful investigation. It is gratifying to know that foreign chemists, generally, have a high appreciation of the work of the association, and recognize its reliability and worth.

An important consideration for the securing of uniform results in our official work is uniform legislation in matters pertaining to fertilizer control. In Alabama the constitution of the State has this provision, adopted before the present Department of Agriculture was established: "No State office shall be continued or created for the inspection or measuring of any merchandise, manufacture, or commodity." No sampling, therefore, or inspection can be done without the consent of the owner and in accordance with his wishes. Neither the commissioner of agriculture nor the State chemist can enforce rules for taking samples. A special law was enacted by the last legislature requiring the commissioner to collect samples of each and every brand of fertilizers sold in the State and have the same analyzed by the State chemist and the results published for the information of farmers and others interested. These samples, coming from first hands, are naturally in fine condition and almost invariably come up to the guaranties, but the farmer has no means of knowing whether the goods delivered some months afterward are the same as the original without another analysis, which he, as well as every other purchaser, has a right to demand free of cost. Complaints are occasionally made that these analyses are not concordant; and how can it be otherwise, unless there be control by a competent chemist in the manufactory, and intelligent oversight in compounding and mixing the materials. and especially very great care in sampling the manufactured products.

It may be said that our work is scientific and above such legal and commercial considerations as those mentioned. It would save much annoyance and leave more time for scientific work in the improvement of methods and the pushing forward of original investigation if the worry of repetitions and revisions could be avoided by the adoption of judicious, uniform legislation and strict conformity thereto by all parties interested.

The composition of soils, of fertilizers, foods, feeding stuffs, dairy products, and other materials connected with agricultural industry and their adaptation to each other, are studied with increasing interest year after year, and the corps of workers in each State are adding to the productive capacity of the soil and introducing intelligent control into the diversified operations of the farm. The increasing demand for fertilizers in connection with a close study of plant growth has led within a few years past to interesting discoveries and should greatly stimulate research, as it doubtless has. Of these recent discoveries of especial interest to the agricultural chemist, none has aroused more enthusiasm than the investigation and final unfolding of nature's method of converting atmospheric nitrogen into a form available for plant food.

That some kinds of vegetation, such as peas, clover, and other leguminous plants, have the power directly or through some intermediate agency to accumulate this element has long been known, but a satisfactory explanation of the process is a late discovery. If the work of microörganisms which produce these results can not be repeated under conditions that will enable the manufacturer to accumulate nitrogenous compounds for practical use in the compounding of fertilizers, it points out to the agriculturist a sure method for collecting this important element in his soil, independently of the manufacturer, that is, by growing leguminous plants and turning their accumulated store of nitrogen into the soil for succeeding crops.

The question of converting atmospheric nitrogen into a form capable of serving as plant food at a moderate cost without the aid of living organisms has not been solved, but who can say that it is not within the range of possibility, and, indeed, on the verge of solution. Dr. Lepel, in a late number of the Annalen der Physik und Chemie, has a paper on the oxidation of nitrogen by means of the electric spark, in which he expresses the hope, justified by extensive observation and experiment, that by means of high voltage discharges, a new method of producing nitric acid from the atmosphere on a large scale may be devised, giving us a source of nitric acid independent of the nitrates.

A more practical discovery for immediate benefit to the farmer is that of immense deposits of phosphates on our Gulf coast. The South Carolina deposits have been taxed to their utmost to supply the demand for commercial fertilizers, and fears were entertained a few years ago that these deposits would soon become exhausted or their value so enhanced as to place them beyond the reach of the farmer, when, opportunely, the discovery was made of immense phosphate beds which are now known to underlie much of the southwestern portion of Florida. These nodular amorphous phosphates are just coming fairly into notice. A recent work on the Phosphates of America, by Dr. Wyatt, has called renewed attention to their great extent and value.

While the existence of phosphatic deposits in Florida to a limited extent has long been known, the first mining operations were commenced in Peace River in 1888. In the following year the Peace River Phosphate Company and the De Soto Company commenced operations by dredging the river, and since that time nearly a hundred companies have been organized, with capital stock ranging nominally from $50,000 to $5,000,000.

Speaking of several counties in southwestern Florida, Dr. Wyatt says: "This section of Florida is virtually underlaid with a nodular phosphate stratum of a thickness varying from a few inches to 30 feet, and covered with an overburden that may be fairly averaged at about 8 feet." A visit was made to some of these localities during the past winter and samples collected for analysis. The deposits examined were of two varieties, which may be denominated hard and soft. The hard variety consists of bowlders of moderately hard rock, some of immense size, cemented to-

gether with white clay, and, as Dr. Wyatt says, sometimes in the form of a white or friable mass resembling kaolin, and probably produced by the natural disintegration of the hard rock by rolling, attrition or concussion. The actual thickness of the deposits is too variable to be computed with any accuracy into an average, but a depth of 20 or 30 feet is not uncommon, and even 50 feet in thickness has been found. The Glaser method used in the determination of iron and alumina gave, for the average of a number of air-dried specimens of the hard variety, from 2 to 4 per cent; of the soft deposit, from 4 to 9 per cent. The complete results are as follows:

For the hard variety:
- 1 to 1½ per cent of moisture.
- 2½ to 10 per cent insoluble matter.
- 2 to 4 per cent oxides of iron and aluminum.
- 35 to 38 per cent phosphoric acid.

Equivalent to ..... 76.30 to 82.84 per cent tricalcium phosphate.

The soft variety gave:
- 2 to 3 per cent of moisture.
- 8 to 35 per cent insoluble matter.
- 4 to 9 per cent oxides of iron and aluminum.
- 22 to 34 per cent phosphoric acid.

Equivalent to ..... 48 to 74 per cent tricalcium phosphate.

Dr. Wyatt gives the following average results of a large number of samples:

	Calcium phosphate.	Iron and alumina.	Silica and silicates.	CO_2.
	Per cent.	*Per cent.*	*Per cent.*	
Bowlders (carefully selected, 120 samples)	80.49	2.25	4.20	2.10
Bowlders and débris (237 samples)	74.90	4.19	9.25	1.90
Soft white phosphate (148 samples)	65.15	9.20	5.47	4.27
Pebbles from Peace River (84 samples)	61.75	2.90	14.20	3.60
Pebbles from drift beds, Polk County (92 samples)	67.25	3.00	10.40	1.70

As some, especially foreign manufacturers, object to buying phosphates which contain over 3 per cent of oxides of iron and aluminum, large quantities of these materials have accumulated at the mines. A few manufacturers, aware of the agricultural value of South Carolina floats, have established mills in Florida for pulverizing these soft aluminous deposits, which are sold to farmers for use without conversion into soluble phosphate. Experiments are now in progress on the Alabama Experiment Station, under control of the chemist, to determine the chemical composition and agricultural value of these soft phosphates when used alone with cotton seed and with cotton-seed meal. If decomposing organic matter, as is believed, renders insoluble phosphates available as plant food to any considerable extent, the question of cheap phosphates will be solved and the farmer enabled to purchase fertilizers at a much less cost than at present. That so desirable a result may be reached is the wish of all who are interested in developing the great agricultural industry of our country.

The president called for the report on phosphoric acid, but it was

ascertained that no reporter was yet present on any of the subjects preceding dairy products.

Mr. Babcock, therefore, was called on to read his report on dairy products.

REPORT ON DAIRY PRODUCTS.

By S. M. Babcock.

At the last meeting of this association the reporter on dairy products was directed to investigate the following questions bearing upon the estimation of the volatile fatty acids in butter: (1) Does the saponification of fats by means of glycerol[1] and caustic soda, as proposed by Drs. Leffman and Beam, of Philadelphia, give identical results with the present official method? (2) Are the results with a block-tin and glass condenser the same? (3) Is it necessary to melt the free fatty acids before distillation? He was also directed to report upon methods for cheese analysis.

In order to facilitate this work a circular letter was issued early in March, describing the methods to be pursued and asking the coöperation of chemists in testing them. No samples were sent, as from the nature of the work it was thought that more satisfactory results would be reached by having the tests cover as great a variety of material as possible. But few chemists have found time to assist in the proposed work; still, I believe from the general agreement of the results that the conclusions reached are approximately correct.

THE GLYCEROL-SODA METHOD FOR THE SAPONIFICATION OF FATS IN ESTIMATING VOLATILE FATTY ACIDS.

This method, as described in the Analyst for August 1, 1891 (p. 153), is as follows:

"The saponification is effected by a mixture prepared by adding 25 cc. of a clear 50 per cent solution of sodium hydroxid to 125 cc. of pure glycerol, and boiling for fifteen to twenty minutes to evaporate the greater portion of the water.

"About 5 grams of the clear fat are weighed out in a flask in the usual manner, 10 cc. of the alkali-glycerol added, and the flask heated over a Bunsen burner. The mixture may foam somewhat. This may be controlled and the operation hastened by shaking the flask. When all the water has been driven off the liquid will cease to boil, and if the heat and agitation be continued for a few moments complete saponification will be effected, the mixture becoming perfectly clear. The whole operation will require less than five minutes. The soap is then dissolved in 90 cc. of water. The first portions of water should be added drop by drop and the flask shaken between each addition in order to avoid foaming. When the soap is dissolved, 50 cc. of diluted sulphuric acid (25 cc. of the concentrated acid to the liter) are added, a piece of pumice dropped in, and the distillation conducted as usual. Blank experiments have given the authors a distillate requiring from 0.2 to 0.3 cc. decinormal alkali.

"The alkali-glycerol is quite viscid when cold. It should be kept in a flask closed with a rubber stopper, and heated when the measured portion is to be taken out."

In a later communication in the Analyst (April, 1892) the authors stated that the boiling of the glycerol-soda solution to remove the water was unnecessary, as the water could be quickly driven off during the saponification. This modification has

[1] Note.—In the spelling of chemical terms this bulletin conforms in the following particulars to the report made to the American Association for the Advancement of Science at its fortieth meeting by the committee on spelling and pronunciation (see Proceedings of the Association for 1891, pp. 175, 176):

Names of chemical substances having the termination **-ide** drop the final **e**, as chlo′rid, i′odid, hy′drid, o′xid; and those having the termination **-ine** drop the final **e**, as chlo′rin, bro′min.

The termination **-ol** is used exclusively for alcohols, as glycerol, not glycerine.

the advantage of giving a solution sufficiently fluid to be measured without previous heating. The same volume of the mixture is used for the saponification, which requires but a minute or two longer than when the concentrated solution is used.

In response to my request for coöperation, reports upon the comparative merits of the two methods were received from the following, viz: The Agricultural Department at Washington, work done by Mr. K. P. McElroy; Colorado Agricultural Experiment Station, by David O'Brine; Delaware Agricultural Experiment Station, by C. L. Penny; Iowa Agricultural Experiment Station, by W. H. Heilman; Maine Agricultural Experiment Station, by J. M. Bartlett; Wisconsin Agricultural Experiment Station, by S. M. Babcock.

Brief summaries of the reports from these chemists giving their opinions of the new method are appended.

Mr. McElroy, of the United States Department of Agriculture, reports that the Leffman and Beam method impressed him unfavorably. "Slight variations in heating made comparatively great differences in the Reichert number. This may be due to the fact that glycerol heated with caustic alkali breaks up into acetic and formic acid, hydrogen being evolved (Watt's Dictionary, old edition, Vol. II, p. 889):

$$C_3H_8O_3 + H_2O = C_2H_4O_2 + CH_2O_2 + 2H_2.$$"

No figures by the two methods are given.

Mr. David O'Brine, of the Colorado Experiment Station, says: "I have tried butter by the Leffman and Beam process as compared with the present process (Bulletin 31, p. 195). The average of five trials by the new method was 2 cc. of decinormal alkali lower than by the present method. The official method gives me 26.01 cc. of alkali for 5 grams of butter fat."

Mr. Penny, of Delaware, gives the result of twenty-six determinations of volatile fatty acids, all of the same sample of butter. Eleven of these determinations were made by the official method, the remainder by the glycerol-soda method of Leffman and Beam. The distillation was made in some cases with a glass condenser, in other cases with a block-tin condenser. In all except four trials made by the glycerol-soda method the free fatty acids were melted previous to distillation.

The following table gives the number of cubic centimeters of decinormal barium hydrate required for 5 grams of butter fat:

Official method—fatty acids melted before distillation.		Leffman and Beam method—fatty acids melted before distillation.	
Glass condenser.	Block-tin condenser.	Glass condenser.	Block-tin condenser.
28.87	28.52	28.57	28.92
28.92	28.87	28.64	28.73
28.84	28.55	28.67	28.72
28.59	28.66	28.66	28.63
	28.73	28.74	28.87
	28.60		28.99
	28.39		
		Fatty acids not melted before distillation.	
		28.83	28.71
		28.85	28.34

Mean of 11 estimations by the official method 28.69
Mean of 15 estimations by the Leffman and Beam method 28.72
Mean of 11 estimations with glass condenser 28.74
Mean of 15 estimations with block-tin condenser 28.68
Mean of 22 estimations with previous melting of fatty acid 28.71
Mean of 4 estimations without previous melting of fatty acid 28.68

Mr. Penny adds: "Other estimations in the case of beef tallow give by alcohol-soda method 0.4 cc.; by the glycerol-soda method, 0.35 cc.; in the case of lard, 0.5 cc. by the former, 0.3 cc. by the latter. Oleomargarine shows a like agreement. Tallow appears to be quite as easily saponified as butter fat by the glycerol-soda method. The time of saponification and distillation by the glycerol-soda method, and without previous melting of the fatty acids, need not exceed forty minutes."

Mr. Bartlett, Maine, says regarding the Leffman and Beam method: "I am much pleased with the method and think it promises to be much better than the old one."

His results with six samples of butter are as follows, expressed in cubic centimeters of decinormal barium hydrate:

	Official method.	Leffman and Beam.
No. 1	27.5	27.1
No. 2	31.4	31.4
No. 3	30.7	30.4
No. 4	30.1	30.3
No. 5	32.1	31.8
No. 6	24.6	24.5
Average	29.4	29.25

Prof. Patrick, of Iowa, writes: "Of the test work proposed by you we have found time in this laboratory only for that on volatile acids, namely, the comparative determination of volatile acids by the association method (Wollny's) and the glycerol method of Leffman and Beam. The work was executed by one of my assistants, Mr. W. H. Heilman, to whom please give credit for it in your report. Three different samples of butter were used.

"(1) A fresh butter, made only two days before being tested.

"(2) A rancid butter, five months old; had been kept in the laboratory during that time.

"(3) A rancid butter; was rancid when received at the laboratory two months prior to date of analysis.

"Results are calculated in cubic centimeters to exactly 5 grams of fat.

Number of sample.	Association method.		Glycerol method.	
		Mean.		*Mean.*
Sample 1	25.63	25.60	25.54	25.49
	25.57		25.44	
Sample 2	24.80	24.80	23.97	23.94
	24.80		23.91	
Sample 3	27.58	27.62	26.52	26.61
	27.66		26.70	

"On the rancid samples the results were sensibly lower by the glycerol than by the association method."

My own work at the Wisconsin Experiment Station was confined to four samples of butter, described as follows:

No. 1, a perfectly fresh butter about one week old, made at the station farm.

No. 2, fresh butter, purchased in town.

No. 3, rancid butter about five months old.

No. 4, rancid butter about one year old.

Eight determinations were made with each butter, four being by the official method and four by the Leffman and Beam method. Two of the tests by each method were

distilled with a glass condenser and two with a block-tin condenser. In these tests the contents of the distilling flask was kept near the boiling point by a low flame until the emulsion was practically broken up, although the fatty acids were not always melted to a clear layer before the distillation was commenced. With the Leffman and Beam method this required scarcely any time, in some cases the fatty acids being melted to a clear liquid within five minutes. With the official method the temperature was kept near the boiling point for about thirty minutes before distillation was commenced.

The results, expressed in cubic centimeters of decinormal alkali, are given below:

Number of sample.	Official method.				Leffman and Beam.			
	Glass condenser.		Tin condenser.		Glass condenser.		Tin condenser.	
		Av'ge.		*Av'ge.*		*Av'ge.*		*Av'ge.*
Sample 1	28.49 28.32	28.40	27.55 27.73	27.64	27.83 28.10	27.96	27.50 27.55	27.52
Sample 2	28.05 27.77	27.91	27.77 28.16	27.96	27.94 28.05	27.99	27.50 27.51	27.50
Sample 3	26.51 26.62	26.56	26.16 25.63	25.89	23.32 23.43	23.37	22.60 22.93	22.76
Sample 4	34.32 34.92	34.62	34.87 35.20	35.03	32.94 33.11	33.02	32.28 32.67	32.47
Average		29.37		29.13		28.08		27.56
General average		29.25				27.82		

Average for glass condenser	28.72
Average for tin condenser	28.34

My first impressions regarding the new method were very favorable to it, as the necessary manipulation is easy and the time required for the saponification of the fat short. Moreover, there is no alcohol to remove, the solution of the soap takes place at once, and the emulsion of fatty acids formed when the soap is decomposed is readily broken up, the acids usually being melted by the time the liquid boils if the heat is applied gradually. All this results in a great saving of time, and I do not think the authors have exaggerated at all in their statement that a determination can be completed in an hour. With two condensers it is easy to make two determinations in an hour and a quarter, counting the time required for weighing the samples.

With fresh butters the results by the two methods agree closely, but with butters which are old and rancid the difference is wide.

It will be observed that with butters 1 and 2, which were fresh, the two methods were not very far apart, although the Leffman and Beam method gave a little lower results. On the other hand, the butters 3 and 4, which were old and quite rancid when the tests were made, gave results which were decidedly too low. During the saponification of these two butters a marked smell of butyric acid was apparent and the low results are undoubtedly due to the escape of free fatty acids which the fats contained before they were combined with the alkali. I attempted to avoid this difficulty by conducting the saponification of the fats at a lower temperature upon a water bath. The addition of about an equal volume of glycerol to glycerol-soda solution makes the saponification easy in this way; at least, a perfectly clear solution is obtained in a short time; but the results obtained in this way were no better than the others.

RESULTS OBTAINED WITH A BLOCK-TIN AND A GLASS CONDENSER.

My experience with glass and tin condensers for this purpose is decidedly in favor of glass. The tin condenser which I used in this work was thoroughly cleaned before my work commenced. The first distillation that was made was from butter No. 1, which had been saponified by the official method. The distillate was quite opalescent through the suspension of solid particles in it which could not be removed by repeated filtrations. It required for neutralization only about 18 cc. of decinormal alkali. The second determination which was made was about as turbid as the first and required 22.5 cc. of alkali. The third and fourth distillations are those given in my report, viz, 27.55 and 27.73, which, on an average, is nearly 0.8 cc. lower than was obtained in a glass condenser. The distillates with a tin condenser were always slightly opalescent and could not be filtered clear. After several distillations had been made in succession the results with a tin condenser agreed quite closely with those in glass, but on the average were a trifle lower even then. Wherever the tin condenser was allowed to stand for a day or two without use the first distillation afterwards was, in every case, decidedly lower than it should have been—in one case, with butter No. 2, only 19 cc. being required. At first I supposed that these discrepancies were due to carelessness on my part, but I am convinced from their number that it is inherent in the method and that the volatile fatty acids act upon the tin and are partially neutralized. It seems probable that the tin finally becomes coated with an insoluble salt which partially protects it from further action.

No comparative tests were made to determine whether it was necessary to melt the free fatty acids before distillation.

The average results reported by those who have undertaken this work are grouped together in the following table:

Comparison of official with Leffman and Beam method for the saponification of fat in the estimation of volatile fatty acids.

Analyst.	State.	Official method.	Leffman and Beam method.
		cc.	*cc.*
David O'Brine	Colorado	26.01	24.01
C. L. Penny	Delaware	28.69	28.72
W. H. Heilman	Iowa	26.01	25.35
J. M. Bartlett	Maine	29.40	29.25
S. M. Babcock	Wisconsin	29.25	27.82
Average of all		27.87	27.03

Result of distillations in glass and tin condensers.

Analyst.	State.	Glass condenser.	Tin condenser.
		cc.	*cc.*
C. L. Penny	Delaware	28.74	28.68
S. M. Babcock	Wisconsin	28.72	28.34
Average		28.73	28.51

The wide variations shown between the present and the Leffman and Beam methods, when applied to some butters should exclude the latter from the official methods. I believe, however, that owing to its greater convenience and ease of manipulation the new method has a value as a preliminary test for adulterations. As its errors appear to be in the direction of too low rather than too high results no mistake would be likely to arise from this. Final judgment, however, in suspected cases,

should only be based upon an examination by the official method. Moreover, the official methods should always be used in scientific work, involving the amount of volatile fatty acids in fats, in order that comparative results may be obtained.

NECESSITY OF MELTING FREE FATTY ACIDS BEFORE DISTILLATION.

In regard to the necessity for melting the fatty acids before distillation the work is not conclusive, as only two trials have been reported, both of which were in connection with the Leffman and Beam method, in which, as has already been stated, the emulsion of fatty acids is broken up very soon after the boiling point is reached. Under such circumstances it is doubtful if the same conditions would hold when the official method was used, and I think it would be unwise to omit this precaution until more data are at hand. I believe, also, that more trustworthy results will be obtained with glass condensers than with block-tin condensers.

I would therefore recommend that the present official method for the estimation of the volatile fatty acids of butter remain unchanged with the exception that in the directions for distillation a glass condenser be specified, and that the following sentence be added to the directions for the titration of the volatile acids, viz: "The number of cubic centimeters of decinormal barium hydrate required by the 100 cc. titrated shall be increased by one-tenth to represent the amount of volatile acids from 5 grams of butter fat."

THE ANALYSIS OF CHEESE.

The analysis of cheese is beset with peculiar difficulties not met with in other dairy products. In the first place, the different constituents are unevenly distributed throughout the mass of the cheese, making it almost impossible to obtain a representative sample. This is owing partly to the evaporation of water from the surface and partly to the fact that changes in the solids, due to the ripening of the cheese, are different at the surface from those in the interior of the cheese. Moreover some of the small cavities of the cheese are often filled with water or whey, while others, especially in very rich cheese, may contain considerable fat. Consequently the composition of cheese depends to a considerable extent upon what portion of the cheese is taken for analysis. When the cheese can be cut it is probable that a narrow sector would nearly represent its average composition. But in the majority of cases the sample must consist of one or two plugs taken with a tryer from different parts of the cheese. This is especially the case in cheese investigation where it is necessary to keep the cheese as nearly intact as possible for future examination. It is desirable, therefore, that a uniform system for taking plugs for analyses be adopted.

Nor do the difficulties end when the sample is secured, for the structure of cheese and the character of its constituents are such as to greatly complicate the furthe course of analysis. The colloidal nature of casein, combined, as it is, with a large amount of fat, interferes seriously with the drying of the cheese at ordinary temperatures, while the volatile compounds formed during the ripening process and the easy oxidation of the fats, and probably other constituents, make results obtained by drying at high temperatures very questionable. Besides, the complete removal of fat from the dried product by the usual methods is almost impractical.

The field for investigation was so wide, and the available data so limited, that it seemed folly to attempt to cover the whole ground in a single year.

I have, therefore, in my circular letter asking for coöperation in this work, confined the tests to be made to a comparison of results obtained by drying cheese at ordinary temperatures over sulphuric acid, and by heating to 100° and, in the estimation of fat to the extraction of the dry mass obtained in the water determination with ether, compared with the method of Short, in which the cheese is ground up with anhydrous copper sulphate and extracted immediately.

Tests of these methods were made at the U. S. Department of Agriculture, at Washington, by K. P. McElroy; at the Connecticut Agricultural Experiment Station, by

Mr. A. L. Winton; at the Nebraska Agricultural Experiment Station, by Mr. T. L. Lyon; at the Minnesota Agricultural Experiment Station, by Harry Snyder, and at the Wisconsin Agricultural Experiment Station, by S. M. Babcock.

These analysts report as follows upon the different tests:

Mr. McElroy, of the U. S. Department of Agriculture, says: "The $CuSO_4$ method for fat in cheese, when tried on a cream cheese, gave higher results than were obtained by direct extraction, after drying the cheese a month over sulphuric acid (33.42 per cent as against 32.50). The manipulation by the former method was peculiarly easy. Drying at 100° to constant weight gave 37.15 per cent of water in the cheese as against 36.07 by drying over sulphuric acid at common temperatures for a month (June 15 to July 15)."

Mr. Winton, of the Connecticut Experiment Station, reports as follows:

Loss at 100°.

The quantity of cheese dried was in each case 5 grams, and was contained either in a flat bottom platinum dish 2 inches square or else in a nickel dish with a circular flat bottom 1⅞ inches in diameter. The final result obtained was found to be the same with both kinds of dishes, although the rate of loss during the first part of the heating was somewhat slower when the nickel dishes were used, owing to the smaller area and greater thickness of their bottoms. In the accompanying table are given the percentages of loss in periods of six hours each up to the forty-eighth hour, and afterwards in periods of twenty-four hours each. The results where nickel dishes were used are not included.

Table showing loss of weight in cheese at 100° for different times of heating.

Time of heating.	Full cream (1).	Full cream (2).	Skim milk (1).	Skim milk (2).	Full cream (old).	Neufchatel (American).	Fromage de Brie (American).	Imitation old English, 3 years old.	Pine apple (yellow) 4 months old.	Pine apple (white) 8 months old.	Pine apple (yellow) 16 months old.	Pine apple (yellow) 5 years old.	Roquefort (imported).
	Pr. ct.	*Pr. ct.*	*Pr. ct*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*
1st–6th hour	35.00	34.00	51.09	52.50	28.27	56.00	60.20	19.08	29.18	26.39	24.01	10.83	38.98
6th–12th hour	1.47	1.20	.87	.48	.92	.82	.00	.71	1.45	.98	.89	.29	.46
12th–18th hour	.32	.32	.20	.07	.24	.35	.....	.35	.06	.44	.40	.09	.46
18th–24th hour	.10	.15	.06	.04	.12	.09	.....	.12	.12	.23	.13	.18	.40
24th–30th hour	.00	Gain*	Gain*	Gain	.10	.02	.....	.12	.00	.15	.07	.05	.40
30th–36th hour	.....	Gain	Gain*	Gain	.09	.....	.....	.12	.00	.15	.03	.09	.20
36th–42d hour	.....	.....	.....	.....	.08	.....	.....	.12	.....	.10	.00	.09	.20
42d–48th hour	.....	.....	.....	.....	.00	.....	.....	.12	.....	.00	.00	.00	.18
48th–72d hour	.....	.....	.....	.....	.....	.....	.....	.48	.....	.00	.....	.00	.66
72d–96th hour	.....	.....	.....	.....	.....	.....	.....	.38	.....	.....	.....	.....	.48
96th–120th hour	.....	.....	.....	.....	.....	.....	.....	.32	.....	.....	.....	.....	†.40
120th–144th hour	.....	.....	.....	.....	.....	.....	.....	.32	.....	.....	.....	.....	.....
144th–148th hour	.....	.....	.....	.....	.....	.....	.....	†.32	.....	.....	.....	.....	.....

* Becoming darker color.

† Drying discontinued.

It will be seen that after persistent heating constant weight was secured in all cases but two, viz, Roquefort and imitation Old English. The sample of Roquefort gave off a strong odor all through the drying, and there is a strong probability that the loss was not water alone. The imitation Old English cheese was hard and horny, and the lumps refused to melt at 100°. Possibly if the material had been previously obtained in a finely divided condition the drying would have proceeded with more rapidity and a constant weight finally secured.

Loss at 110°.

The samples of imitation Old English cheese and five-year-old pineapple cheese were dried in an air bath provided with a gas regulator which kept the temperature at 110°. The drying was discontinued after the thirty-second hour. Following are the results:

Time of heating.	Imitation Old English.	Pineapple, 5 years old.
	Per cent.	*Per cent.*
2 hours.	20.48	10.64
6 hours.	.87	1.25
6 hours.	.41	.40
6 hours.	.44	.54
6 hours.	.31	.37
6 hours.	.27	.36
32 hours.	22.81	13.56
Loss at 100° C. in 32 hours	20.42	11.44
Loss at 100° C. in 170 hours	22.56	.....
Loss at 100° C. in 42 hours		11.62

Undoubtedly the loss at 110° represents something besides water, as a very disagreeable odor was given off at that temperature.

"Loss over sulphuric acid.

"A thin slice of the sample of 'full-cream cheese (1),' weighing 6.1323 grams, was put in a desiccator over strongest sulphuric acid March 30, the loss in weight being determined from time to time. At the date of writing, June 30, the material is still losing in weight, although at a very slow rate, and it may be months before the last traces of moisture are removed. The following table shows the percentage of loss at first from day to day, afterwards in periods of from three to eighteen days.

Time of standing, in days.	Percentage of loss.
1	17.82
1	8.90
1	2.80
1	1.03
3	1.88
3	.83
3	.53
3	.40
3	.27
3	.13
6	.26
6	.18
6	.15
6	.11
6	.09
9	.08
*17	.11
18	.10
96	35.67
Total loss at 100	36.89

*Acid boiled.

"Fat.

"Fat was determined, in all of the samples used in the experiments on determination of water, by the method proposed by Short, described in your circular. The ground mixture of cheese and anhydrous copper sulphate was dry and pulverulent and readily gave up its fat to ether. The extraction in each case was carried on in successive periods of four hours each. In all the samples, with one exception, the fat extracted between the fourth and eighth hour was less than 0.4 per cent, and between the eighth and twelfth hour was practically nothing. The imitation old English required a somewhat longer extraction, the fat removed between the fourth and eighth hour amounting to 0.66 and between the eighth and twelfth hour to 0.27. At the end of the twelfth hour the extraction of even this sample was complete. Duplicate determinations agreed sharply. No opportunity was presented to compare results by this method with those obtained by extracting the material dried over sulphuric acid, or at 100°. A single trial with the plan proposed by Prof. W. W. Cooke, of extracting the cheese without grinding, drying, or mixing with dehydrating substance, was unsatisfactory because of the slowness with which extraction proceeded. The fat not being removed completely after twenty-four hours, the trial was abandoned. The reason for this slowness of the extraction was probably more because the cheese was not in a finely divided condition than because of the presence of moisture."

Mr. Lyon, of the Nebraska Experiment Station, reports:

"I herewith report results of analysis of a sample of cheese according to the methods called for by the reporter on dairy products for the Association of Official Agricultural Chemists.

"The sample was of a cream cheese, and was, according to the dealer, about two months old.

"All results are of duplicates, except the determination of moisture by drying over sulphuric acid. Squibb's 'stronger ether' was used for the extraction. The ether extract from the mixture of cheese and anhydrous copper sulphate had a pale-blue tinge. For both extractions a siphon extractor was used.

"Determination of moisture in cheese by standing over sulphuric acid—

Weight of cheese placed in desiccator on May 13 ..... 5.3053 grams.

Per cent loss by drying over H_2SO_4:

	Per cent.
To May 27	36.61
To May 31	36.71
To June 2	36.73
To June 3	36.74

"Determination of moisture in cheese by drying for twelve hours at temperature of boiling water—

	I.		II.	
Weight of cheese taken	5.7922	grams.	5.5724	grams.
Loss of cheese by drying at 100°:				
After 4 hours	31.69	per cent.	29.02	per cent.
After 8 hours	35.46	per cent.	34.37	per cent.
After 12 hours	36.92	per cent.	36.93	per cent.

"Determination of fat in cheese by ether extraction of a mixture of about equal parts cheese and anhydrous copper sulphate—

"Extraction continued fifteen hours.

	Per cent.
Determination 1 gave	34.39
Determination 2 gave	34.47
Average	34.43

"Determination of fat in cheese by extracting with ether the cheese dried by standing over sulphuric acid—

"Extraction continued fifteen hours.

	Per cent.
Determination 1 gave	33.93
Determination 2 gave	33.98
Average	33.95

"The above results were, of course, obtained upon the same sample of cheese."

Mr. Snyder, of the Minnesota Agricultural Experiment Station, says, in regard to his work: "The results given are averages of duplicate determinations, the duplicates by the copper sulphate method agreeing as well as those obtained by extracting the dried mass." The following table gives results of his work:

No.	Age of cheese.	Water.		Number of days over H_2SO_4.	Fats.	
		100°.	Over H_2SO_4.		Short's.	Mass extraction.
		Per cent.	*Per cent.*		*Per cent.*	*Per cent.*
1	6 months	34.86	33.66	46	33.16	32.98
		34.45	33.23			
2	5½ months	33.59	32.30	22	34.00	34,01
		32.97	32.26			
3	5 months		34.15	20	26.00	26.58
4	6 months		30.10	20	36.26	36.24
5	4½ months		35.25	20	35.04	34.78
6	5 months		27.65	20	46.10	46.83
7	6 months		30.75	20	36.30	36.63
8	6 months		30.40	20	45.18	45.25
9	6 months		30.10	20	45.50	45.32

The work in Wisconsin has included the tests specified in my circular letter, and in addition I have made determinations of fat by the Babcock method for the estimation of fat in milk, as described in Bulletin 24 of the Wisconsin Agricultural Experiment Station. Five different cheeses, designated as follows, were examined:

No. 1, full-cream cheese, 15 months old.
No. 2, full-cream cheese, 18 months old.
No. 3, full-cream cheese, 16 months old.
No. 4, full-cream cheese, 4 months old.
No. 5, full-cream cheese, 4 months old.

In the water determination the duplicate samples from Nos. 1 and 2 were from different plugs taken from the same portion of the cheese as near together as was practical, and I believe are of as nearly the same composition as can be obtained from different plugs. With No. 3 several plugs were taken and all chopped together until the particles were quite small; the portions for all of the estimations were then taken from the mixed mass, so that all of the samples must have been practically alike. With Nos. 4 and 5 the duplicate samples were from the same plug, a thin slice from it being placed alternately in the two watch glasses in which the cheese was dried. From 7 to 10 grams of cheese were taken for each determination.

The results are given below:

Table showing comparative loss in weight in cheese by drying over sulphuric acid at ordinary temperatures and by drying at 100°.

No. of cheese.	Dried over sulphuric acid.				Dried at 100°.			
	Time (days).	Loss (per cent).			Time (hours).	Loss (per cent).		
		1.	2.	Average.		1.	2.	Average.
No. 1	10	27.06	28.14	28.68	2	25.09	25.05	30.09
	20	27.36	28.64		5	28.60	28.52	
	30	27.80	.....		6	28.49	29.61	
	45	28.01	29.36		60	29.68	30.60	
No. 2	45	25.29	24.48	24.88	3	23.80	23.69	25.78
					4	24.57	24.41	
					60	25.96	25.61	
No. 3	11	23.12	22.93	23.40	5	23.76	23.69	25.04
	35	23.48	23.32		6	23.83	23.74	
					22	24.54	24.46	
					62	25.04	25.04	
No. 4	15	28.91	29.14	29.69	5	23.66	22.14	30.65
	30	29.57	29.81		6	23.75	22.24	
					30	29.02	28.23	
					70	30.78	30.52	
No. 5	15	29.75	30.00	30.50	5	25.76	26.85	32.10
	30	30.48	30.53		6	25.87	26.98	
					30	31.22	31.50	
					70	32.05	32.15	
Average of all determinations	.....	.....	.....	27.43	.....	.....	.....	28.73

The duplicate results in all cases, except in the four trials with Nos. 1 and 2 where the samples were taken from different plugs, were very satisfactory. In the four tests in which different plugs were used the average difference between duplicate determinations was .84 per cent, with a maximum difference of 1.35 per cent. In the six tests, in which care was taken to have the duplicate samples as nearly alike as possible, the average difference was only .12 per cent with a maximum of .26 per cent. This illustrates well the point already mentioned, that one of the greatest difficulties in the analysis of cheese is involved in taking the sample. There seems to be no trouble in getting concordant results upon water with either method when the samples tested are alike.

In drying over sulphuric acid a large surface of acid was exposed in the desiccator and the acid was renewed toward the end of the operation. In one or two cases the weights became practically constant, and in no case did the lowest exceed 2 milligrams per day when the cheese was removed.

The samples dried at 100° were heated until they ceased to lose weight, and in some cases there was a slight gain. The lowest observed weight was taken. In all cases the cheese was more or less colored by the heat, and the odor which was continuously given off showed loss of something besides water.

In all cases the loss in the samples heated to 100° was considerably greater than in those dried at ordinary temperature, the average difference being 1.3 per cent. The length of time when the loss at 100° was equal to the total loss over sulphuric acid is not uniform in all cases, it being much less in old well-ripened cheese than in cheese in which the casein is not thoroughly broken down.

ESTIMATION OF FAT.

The fat was determined in all of the samples used for water by extracting the dry residue with ether, and in other samples, taken at the same time as those for water, by Short's copper-sulphate method, and also by Babcock's centrifugal method. The

duplicate results numbered 1 and 2 in the table below correspond with the same numbers in the table of water determination. In tests by Short's method, and also those by Babcock's method, the figures connected by a brace are from tests where precautions were taken to have the duplicate samples as nearly alike as possible. The other tests by these methods are of plugs taken from different parts of the cheese, and are designed to show the variations which may arise from sampling.

The extraction in every case was made with absolute ether and was continued for fifteen hours. The solution of fat was in every case clear, that from the cheese which had been heated being dark colored.

The tests by the Babcock method were made in the cream tubes designed by Mr. Bartlett, of the Maine Experiment Station, the calibration of all the bottles used being carefully tested. The test bottle was first weighed, after which about 8 grams of cheese were put into it and the bottle weighed again. About 15 cc. of hot water were put into the bottles, which were set upon a water bath until the cheese became softened, so that by shaking the bottle a homogeneous emulsion was formed. The test was then completed in the same manner as with milk, the final reading being multiplied by 18 and the product divided by the weight of cheese taken, in grams, for the per cent of fat. A new cheese or a skim cheese will not readily dissolve in this way, but if a few drops of strong ammonia are put into the bottle after the water is added the cheese will soon become softened so that the test can be made. The sulphuric acid should be added slowly with shaking, a little more being used than is necessary for solution.

The results of the tests are given below:

Table showing results of fat determinations in cheese by different methods.

No. of cheese.	Extraction of dried residue from water determination.				Short's method.		Babcock's method.	
	Dried over H_2SO_4.		Dried at 100°.					
		Av'ge.		Av'ge.		Av'ge.		Av'ge.
No. 1	40.01	39.38	38.71	38.09	41.19	40.78	38.83	
	38.76		37.47		40.38		39.16	38.24
							37.72	
No. 2	40.31	40.55	39.56	39.65	40.49	40.45	40.67	40.33
	40.79		39.75		40.42		40.00	
No. 3	*44.19	44.15	*43.32	43.30	*41.59		*44.74	
	*44.11		*43.29		*44.56		*44.41	43.74
					44.71		43.83	
					45.26	44.78	43.34	
					44.01			
					44.14			
					43.98			
					41.47			
No. 4	36.09	35.99	35.06	34.86	36.30	36.20	33.25	
	35.89		34.66		36.10		36.26	
							36.93	36.70
							36.71	
							37.08	
							36.95	
No. 5	36.98	36.90	35.80	35.78	36.69	36.60	37.47	
	36.82		35.77		36.51		37.12	37.14
							36.79	
							36.87	

* These samples as nearly alike as possible.

The amounts of fat found by extracting the mass dried over sulphuric acid by Short's copper-sulphate method and by Babcock's centrifugal method agree quite closely, especially where care was taken to have the samples alike. The mass dried at 100° C. did not, however, give up its fat readily to ether, and in every case the results are considerably lower by this method than any other. As a rule Short's method gives a little higher result than does the extraction of the mass dried at ordinary temperature.

In order to determine whether the low result with the cheese dried at 100° C. was due to the more difficult extraction of the fat from this portion or to the destruction of the fat by oxidation while heated the extracted residues from Nos. 3, 4, and 5 were transferred to the Babcock test bottles and a determination of fat in them made by this method.

The result of the tests is given below:

Table showing fat obtained by Babcock test in the residue of cheese after fifteen hours' extraction with ether.

Number.	Cheese dried over H_2SO_4.	Cheese dried at 100°.
	Per cent.	*Per cent.*
No. 3	.....	.25
		.60
No. 4	.26	1.12
	.25	1.55
No. 5	Trace.	.77
		1.03

The fat found in these tests added to that obtained by extraction in the several cases gives results which agree very closely indeed with the other determinations. All this seems to indicate that the extraction of fat by Short's method is more complete than where the dried mass is extracted, and further that the extraction of fat from cheese that has been heated for a considerable time to 100° is impractical. It also shows that the estimation of fat in cheese by the Babcock method is nearly as satisfactory as by the gravimetric methods.

The average results by the different analysts on water and fat are grouped together in the following table:

Analyst.	Water.		Fat.			
	Over H_2SO_4.	At 100°.	Direct extraction of residue.		Short's method.	Babcock's method.
			Dried over H_2SO_4.	Dried at 100°.		
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Agricultural Department.—Mr. McElroy	36.07	37.15	32.50	.....	33.42	.....
Connecticut.—Mr. Winton	35.67	36.89	.....	.....	.....	.....
Nebraska.—Mr. Lyon	36.74	36.93	33.95	.....	34.43	.....
Minnesota.—Mr. Snyder	32.86	33.96	37.62	.....	37.50	.....
Wisconsin.—Mr. Babcock	27.43	28.73	39.39	38.34	39.72	39.49
Average	33.75	34.73	35.86	.....	36.27	.....

The water determinations show that closely agreeing duplicate results may be obtained by either method, but that heating to 100° until constant weight is reached gives considerably higher results than drying over sulphuric acid at ordinary

temperatures. There are no data to show how much of the difference is due to the removal of other volatile constituents than water. I believe, however, that much of the difference may be attributed to this cause. I had hoped that by heating from four to ten hours there would be found a time where the two methods would practically agree with the majority of samples. Such however is not the case, and in order to obtain perfectly consistent results in all cases we must choose between drying over sulphuric acid and drying at 100° to constant weight. As either of these methods requires so much time and so many weighings, I would suggest that for the present the association adopt as a standard for water determination the loss incurred by ten hours' heating at 100°. It is not probable that much hygroscopic moisture would be retained after this time, and the loss of other matter takes place so slowly that I believe the results obtained in this way would rarely vary either way more than three or four tenths per cent from the actual.

The good opinions expressed by all of the analysts regarding the method proposed by Mr. F. G. Short for the estimation of fat warrants me in recommending its adoption as the official method. For purposes of inspection the Babcock method is much quicker than any of the others, and in general is sufficiently accurate.

Mr. BABCOCK. I think it would be well to have any discussion which may pertain to butter and milk take place before the method of cheese analysis is taken up.

The PRESIDENT. If there is no objection, we will proceed as indicated by Prof. Babcock. I would be very glad if each speaker would announce his name, as it is very difficult to hear in this hall, and I am not always able to tell even the names of some of my old friends. It is suggested that if there are any papers to be read on that subject they be read now, and before we proceed to the discussion. Let us have the papers now, if there be any on the subject. If not, we will proceed with the discussion.

Mr. FREAR. It seems to me that Assistant Secretary Willits should be notified of the opening of the association and his presence requested. I would include Secretary Rusk, if he were not out of the city. I move that a committee of three be appointed to invite Mr. Willits to attend the meetings of the association.

The motion was adopted.

The chair appointed as such committee Messrs. Frear, Robinson, and Voorhees.

Mr. WILEY. I move that we proceed with the discussion of the first part of the paper.

Carried.

Mr. CRAMPTON. I do not know that it is necessary to add anything to the report in reference to the Leffman and Beam method. What I have to offer is in the same direction. Perhaps I might say that this method has been investigated in the laboratory of the Internal Revenue Bureau during this last summer, and with the results at which the reporter arrived. That is, analysis by the Leffman and Beam method has given lower results in all cases than by the official method.

Mr. WOODS. I would like to hear from Mr. Penny about his experience with glass and tin condensers, as it seems that Delaware and Wisconsin disagree on this question.

Mr. PENNY. My experience with the block-tin condenser was that turbidity was not entirely removed by filtration. But the figures obtained show no difference that I can detect.

Mr. HUSTON. I think the difference may be due to the condition of the condenser. While I do not know how the particular substances distilled in that way may affect the block-tin condenser, I do know there is a vast difference in the action of water on the outside of the tin condensers. I have had some tin that in about three weeks was utterly destroyed, while with other block-tin condensers very good satisfaction was given. I think before we reach a final decision in this matter we had better specify the kind of tin we are going to use.

Mr. BABCOCK. The subject of tin has become of such importance that I think we had better take hold of it. The condensers used by me were of the tin such as is used in soda fountains. It is supposed to be pure tin, but in regard to that I do not know. The results obtained by Mr. Short coincide with mine, where he found the first distillations made in the block-tin condensers were very much lower in volatile acids than the same butter gave in a glass condenser. Mr. Short gave no figures bearing upon it. He simply said that he had had the same trouble, and had thought that some samples of what were afterwards found to be pure butter were supposed to be oleomargarine or butterine. At any rate, if there is so much difference in tin, if we have to analyze our condensers, it seems to me it would be better to adopt a plan where it is not necessary to do so, and use glass.

Mr. SCOVELL. Two or three years ago, in some work for the U. S. Department of Agriculture, I noticed the same discrepancy between the block tin and the glass, and we finally adopted the glass tube on account of the discrepancies of the block tin. I will say that the block tin is supposed to be pure, although we did not analyze it. I am therefore decidedly in favor of the recommendation of the reporter, on account of the absolute necessity of correct methods for the association.

Mr. WILEY. I think the block-tin condensers should be excluded from all kinds of laboratory work. Glass tubes I think are much to be preferred, as glass has given much better satisfaction, and I am very glad to hear the recommendation of the reporter that the block-tin condensers be excluded from use in the determination of volatile acids in butter.

The PRESIDENT. We use the glass in my laboratory altogether.

The debate being closed on the first part of Mr. Babcock's report, the president called for the discussion of the second part, relating to cheese.

Mr. SCOVELL. Heretofore we have always chosen to use 100°, or the temperature of boiling water, for drying. I suggest that it be used here.

Mr. WINTON. I found that the sum of water, fat, ash, and nitrogen, multiplied by 6.25, fell short of 100 from 1 to 3 per cent. This deficiency

is usually considered as lactic, butyric acids, etc. I think it is explainable also by error due to the use of improper factors for calculating crude protein.

Mr. PENNY. I think the error lies chiefly in multiplying the nitrogen by 6.25. I find in analyses of casein percentages of nitrogen given as 15.91 and 15.65, making the factors 6.28 and 6.39 respectively. The nitrogenous matter of fresh cheese is not wholly casein, but partly paracasein, which, according to analyses, contains 15.14 per cent of nitrogen, making the factor 6.60.

Mr. BABCOCK. The point raised has received considerable attention by me in my report.

I believe that a great many of the differences come through having wrong factors, but on the whole it seems to me better to retain the factor 6.25, inasmuch as it is held in all other determinations of albuminoids. It should be remembered that there is also in cheese a considerable quantity of amide matter, and it seems wrong to include it in this way, especially as there is also in well-ripened cheese quite a large proportion of nitrogenous matter that is present in the form of ammoniacal salts. There seems only one question, whether to adopt the factor 6.36 or adhere to the one adopted in all of our other work, 6.25 for protein.

Mr. SCOVELL. I move the usual form, that we now take up the subject of dairy products; that the reporter read the several sections seriatim, and if there is no objection that the section stand unaltered, and finally that it be adopted as a whole.

Adopted.

Mr. WILEY. All the members are familiar with the processes, and instead of reading the full sections I suggest that they be read by title only.

The various methods for the analysis of dairy products were then read in full or by title, and adopted as amended by the reporter.

Mr. WOODS. I suppose this association is ready to adopt these methods on cheese. We are taking methods that have never been investigated by the association as an association, and propose to adopt them without trial. Would it not be better to make recommendations for further trial of this method, and not commit ourselves to a process that in two or three years we might wish to change?

I think the greatest criticism that can be brought upon us as an association is that we have introduced methods of analysis without sufficient trial. I move the adoption of the cheese methods not as official, but as recommended for our guidance until such time as the association sees its way to the adoption of an official process of a permanent character.

Adopted.

Mr. HUSTON. Now about the extraction of fat in milk. In the official methods as we apply them we have a number of different kinds of glass

tubes, and I want to ask the reporter if he has any objection to adding the words, "or of some similar design?" I have in my laboratory an extraction tube substantially like the Gooch crucible, except it is glass. I do not see any reason why it would not act as well for the purpose of extracting the fat from cheese. I would like to see that changed so as to include some of the forms already in general use in the laboratory.

Mr. BABCOCK. I accept the suggestion.

Mr. WHEELER. As I understood from the reporter that a factor of 6.33 for casein is used in Europe in the determination of albuminoids in cheese analysis, it occurs to me, inasmuch as a great deal of European cheese is sent here, that it might be best to retain the factor as they have it. I simply offer this as a suggestion.

Mr. FREAR. I beg to report that the committee appointed by this association to wait on Assistant Secretary Willits, and inform him that this association was duly organized and would be pleased to be honored by his presence, have performed that pleasant duty, and that he replied that he hoped to be present at sometime during the present session.

The PRESIDENT. We will be very glad to see him at any time.

Mr. PENNY. I move that the reporter on dairy products for next year be instructed to investigate the comparative merits of drying in air and in hydrogen.

Adopted.

Mr. Wiley called the attention of the association to the many delightful excursions the capital city afforded, and suggested that the association avail itself of one of them. The subject was laid over for further consideration.

The hour of adjournment having arrived, the president announced that the first subject to be considered at the afternoon session would be the report on phosphoric acid, by Mr. Lord.

AFTERNOON SESSION, THURSDAY.

The president called the association to order at 2 p. m.

Mr. Lord presented the following report:

REPORT ON PHOSPHORIC ACID.

By N. W. LORD.

In addition to the regular work of investigating the analytical results obtained by the members of the association with the official methods, the reporter was instructed to consider the question of the addition of ammonium citrate to the wash water in filtering citrate-soluble phosphoric acid (p. 113, Bul. 31); to look into the advisability of adopting the method of De Roode of determining phosphoric acid and

nitrogen in the same weighed portion of sample (p. 111, Bul. 31); to look into the question of the length of time required for the digestion fluid to reach the required temperature of 65° in the determination of citrate-soluble phosphoric acid; also the size and shape of the flasks and the proportions of the water bath (p. 115, Bul. 31).

On March 3, 1892, a circular letter was sent to 52 chemists, nearly all *ex officio* members of this association, asking their coöperation in the analysis of four samples of phosphates. Later samples were sent to all who were willing to undertake the work. By May 15 results had been received from 14 analysts. These were tabulated and sent to all who had reported, with the request that they revise their work where it appeared advisable and seek the causes of variation from the average, and suggesting certain lines of work. A circular had been sent with the samples stating the points on which the reporter was instructed to request special work or information.

The samples selected were as follows:

(1) A mixture containing iron and alumina phosphates (Keystone phosphates) with "ammoniates."

(2) A mixed fertilizer containing guano and tankage, acidified with sulphuric acid.

(3) A pure acidulated bone, containing 1,750 pounds steamed bone tanking and 250 pounds of 60° sulphuric acid.

(4) A basic slag, an old sample we had had on hand for some time.

These represent classes of goods which make up a large part of our Western trade and cause much trouble to the analyst.

The samples were spread out on trays and air-dried till "dusty," but subsequent experiments showed that they still lost moisture upon exposure and a third circular was sent to all the chemists who had received samples, calling their attention to the fact and urging caution in handling.

Several pounds of each goods were sifted and ground in an iron mortar until the whole was put through a 25-mesh brass-wire sieve, the powdered sample again repeatedly mixed by hand, and put through coarse sieves until a most thorough mixture was obtained, then 30 3-ounce bottles were filled by putting small portions successively into each bottle, repeating this till all were filled; all were immediately corked and sealed with wax. The reporter feels that the samples were as absolutely uniform as the nature of the substance could possibly permit.

Table No. 1 gives the final results received from the analysts reporting upon the samples. I have changed the method of comparing results with the mean; the errors are not functions of the amount of the ingredient determined, but of the process and manipulation; hence to calculate their percentage on the amount found is misleading. I have calculated the percentage of all the results which come within certain limits of variation from the mean, which is a better measure of the accuracy of the method or the analyst as it leaves out the amount present, which has nothing to do with such matters as solubility of precipitates, failures to completely dissolve, etc.

Following the table I give the notes, comments, and experimental work of each analyst reporting, adding such suggestions of my own as may seem necessary.

(1) *U. S. Department of Agriculture.*—Reported by H. W. Wiley. The analytical work done by Mr. K. P. McElroy. Methods strictly official. Total P_2O_5 in samples 1, 2, and 3 determined by Burney's H_2SO_4 method. Sample No. 4 dissolved in HNO_3. Factor for $Mg_2P_2O_7$ to P_2O_5 0.6396. Ammonium citrate neutral by $CaCl_2$ method. A sample of the solution used was sent with the report, which by our test ($CaCl_2$ method) was very slightly more acid than that we had used and considered neutral.

(2) *Pennsylvania Agricultural Experiment Station.*—Reported by William Frear, State College, Pa. Analytical work done by Mr. A. W. Fries, who furnishes the following comparison of results:

	1.		2.		3.		4.	
		Av'ge.		*Av'ge.*		*Av'ge.*		*Av'ge.*
Moisture:								
Before analysis at 100°.....	3.80 3.87	3.84	11.07 10.92	11.00	6.45 6.41	6.43	.90 .88	.89
After analysis at 100°.....	3.81 3.90	3.86	10.96 10.93	10.95	6.20 6.27	6.24	.75 .78	.77
Total phosphoric acid:								
(*a*) Solution in HNO_3+HCl.....	17.60 17.65	17.63	14.44 14.41	14.43	22.58 22.65	22.62	18.68 18.54	18.61
(*b*) Ignition with SiO_2 and then solution as before.....	17.59 17.72	17.66	14.48 14.44	14.46	22.62 22.59	22.61	18.97 19.07	19.02
Soluble phosphoric acid.....	2.08 2.13	2.11	2.79 2.83	2.81	2.60 2.62	2.61	Trace.	
Insoluble phosphoric acid:								
(*a*) Washed with water at 70°.....	7.92 7.96	7.94	5.50 5.54	5.52	11.09 11.21	11.15	12.03 11.93	11.98
(*b*) Washed with $\frac{1}{10}$ citrate solution at 70°.....	8.01 8.06	8.04	5.55 5.49	5.52	11.04 11.14	11.09	11.90 12.01	11.96

Nos. 1–3 were very hard to filter from citrate solution.

The citrate solution's neutrality was determined by coralline.

The bath for citrate treating is 36 by 14 by 4 inches, with a false bottom of wire netting, 1½ inches from the bottom of the bath.

This bath holds forty 300 cc. Erlenmeyer flasks, such as we use for the citrate. The water in the bath is kept at the level of the liquid in the flasks.

With the bath at 65° the introduction of the flasks causes a drop of little more than half a degree ordinarily, and within one or two minutes the bath is restored to a temperature of 65°. But 100 cc. of either citrate or of water put into the flask at 25° and inserted in the bath requires fourteen minutes to reach 63°, and subsequent exposure for nearly one hour failed to secure any further elevation of temperature. This was not due to discrepancy in the thermometers used, for the test was repeated, using the same thermometers in reverse positions. These results indicate need of very specific directions at this point in the analysis.

(3) *Kentucky Agricultural Experiment Station.*—Results reported and analytical work done by H. E. Curtis, Lexington, Ky. Moistures: two grams dried five hours in water oven 12 by 10 inches, 1 inch water space between walls, thermometer in oven registered 98°. Soluble acid washed till filtrate amounted to 250 cc., using pump and moderate suction. Insoluble acid, official method, residue and filter put in 250 cc. Erlenmeyer flask and stirred with 25 cc. citrate solution till broken up, then the rest of the 100 cc. added—washed with one part citrate to three parts water of 65° C.—residue burned and dissolved in HCl. Total phosphoric acid, determined by H_2SO_4 and HgO. After all weighings were made duplicate moisture determinations on Nos. 2 and 3 gave—

	Per cent.
No. 2	9.59
No. 3	6.53

Total phosphoric acid with HCl and $KClO_3$ on No. 1 gave 17.63 per cent. By De Roode's method, using 1 gram salicylic acid and 5 grams sodium thiosulphate gave—

	Per cent.
No. 1	17.73
No. 2	14.28
No. 3	22.16

The water bath for citrate digestion was square, 14 by 15, 4¾-inch-deep, water stands 1¼ inches deep, covering the bulb of the thermometer. The time required to

reach 63° was about twelve minutes. The solution never reached 65°. The citrate solution was neutral to litmus and also by "Huston's method."

The tabulated results are on the second set received from Mr. Curtis. The first report sent "subject to change" was practically identical except as to total P_2O_5 in Nos. 1 and 2, which were considerably lower, but the method is not stated except that it was "official."

(4) *State Laboratory, Auburn, Ala.*—Reported by Dr. N. T. Lupton. Analytical work done by Mr. R. E. Noble; methods, official. Total phosphoric acid determined after ignition with $Mg(NO_3)_2$; residue dissolved in HCl.

(5) *Clemson Agricultural College, Fort Hill, S. C.*—Reported by Mr. M. B. Hardin. Analytical work by F. S. Shiver and R. N. Brackett, sending separate sets of results.

Moisture determinations at end of work are reported by Mr. Shiver as follows:

No. 1, 3.31; No. 2, 9.69: No. 3, 6.45; No. 4, 0.94.

They also send the following comparative work on different methods for total phosphoric acid:

Total phosphoric acid.

[Analyst, F. S. Shiver.]

	H_2SO_4 and KNO_3	HNO_3 and HCl.	$Mg(NO_3)_2$ HCl and NH_4NO_3	HCl (alone).
No. 1	17.24	17.21	.....	.....
No. 2	13.91	.....	13.74	.....
No. 3	21.76	21.75	.....	.....
No. 4	18.17	.....	.....	18.17

Total phosphoric acid.

[Analyst, R. N. Brackett.]

	HCl and 6 HNO_3	H_2SO_4 and KNO_3	Ignition and solution in HCl
No. 1	16.98	17.21	.....
No. 2	13.64	13.93	13.37
No. 3	21.62	21.56	.....
No. 4	18.00	18.20	.....

(6) *North Carolina Agricultural Experiment Station, Raleigh, N. C.*—Reported by Prof. H. B. Battle. Analytical work done by J. S. Meng and F. B. Carpenter. The total phosphoric acid was determined by solution in HNO_3 and HCl. The results tabulated are the averages of very closely agreeing duplicates for all totals and insolubles.

The 6-ounce Erlenmeyer flasks were used for digestion. The insoluble residue in Nos. 2 and 3 are noted as sticky and hard to wash. The following description of bath is from Mr. F. B. Carpenter:

"The citrate digestion bath used by us is circular in shape, 20 inches in diameter and 5 inches deep. When used this bath is filled to within one-half an inch of the top, making about 64 liters of water. The flasks are held in position by a rack with spring clamps for the top of flask and a zinc plate with circular holes cut in the edge to fit near the bottom of flask. This rack is made to carry twelve flasks at one time, and is kept revolving by a fly-fan propeller, which sits under the bath. With this arrangement there is no difficulty in keeping the water at a uniform temperature of 65° in all parts of the bath. The heat is furnished by Bunsen burners. The rack is so constructed that the flasks are readily taken out for shaking.

"The time it takes for the solution in the flasks to reach 65° will be shown by the following temperatures taken from one of the flasks:

	Degrees.
Temperature when put in	20
Temperature after 5 minutes	60
Temperature after 7 minutes	62
Temperature after 10 minutes	63½
Temperature after 13 minutes	64
Temperature after 15 minutes	64½

"The temperature remained at 64.5° during the next fifteen minutes of digestion, while the water in the bath was 65°.

"My custom is to let the water come up well around the flask, considerably above the solution in the flask. I have found by experiment that there is quite a considerable difference in results according to the depth the flask is immersed in the water. I think this is one point that has never been discussed, but is of considerable importance."

ADDITIONAL NOTES BY J. S. MENG.

"Sample No. 3 is very light, and a portion is lost in transferring from the weighing glass to the flask. In order to test this, I made an analysis in the usual manner, and obtained 21.79 per cent P_2O_5. I then weighed a small covered beaker, added some of the substance, and weighed again. This was treated directly in the beaker, and the results of two analyses were 22.08 per cent and 22.12 per cent. Care was taken in all cases to wash the precipitates until free from chlorids."

(7) *Michigan Agricultural College.*—Reported by R. C. Kedzie, Agricultural College, Ingraham County, Mich. The moisture was determined in an air bath so regulated as to indicate 100° constantly. A steam bath was tried, but 100° was not reached in it. In the tabulated results the moisture found by drying in the steam bath is given; the temperature reached was 95°. The result on water-soluble P_2O_5 varied with the amount of water used, and those tabulated were obtained by washing to 250 cc. The following comparison is given:

	250 cc. wash.	300 cc. wash.
	Per cent.	*Per cent.*
No. 1. Water-soluble P_2O_5	1.90	2.37
No. 2. Water-soluble P_2O_5	2.55	3.28
No. 3. Water-soluble P_2O_5	2 52	3.09

The water bath for citrate-soluble was a granite iron kettle 11 inches in diameter. The introduction of an Erlenmeyer flask with 100cc. citrate lowered the temperature 1°. It required 3½ minutes for the water in the flask to reach 65°.

Comparison of results of washing with water and with dilute citrate solution.

	Washed with water.	Washed with 25 per cent citrate.
	Per cent.	*Per cent.*
No. 1. Insoluble P_2O_5	7.54	7.42
No. 2. Insoluble P_2O_5	5.24	5.11
No. 4. Insoluble P_2O_5	12.60	12.28

Comparative results on total P_2O_5.

	De Roode method unmodified.	Same with zinc and salicylic acid.
	Per cent.	*Per cent.*
No. 1. Total P_2O_5	16.73	16.79
No. 2. Total P_2O_5	13.85	13.75
No. 3. Total P_2O_5	21.51	21.43
No. 4. Total P_2O_5	18.12	.....

(8) *Mississippi Agricultural Experiment Station, Starkville, Miss.*—Reported by L. G Patterson. Analytical work done by Mr. Patterson and Mr. W. R. Perkins. Reported separately. Notes by Mr. Patterson:

"The moisture determinations were made by drying in a watch crystal at 100° in a double-walled steam bath 10 by 12 inches; drying continued for five hours. In the determination of citrate-insoluble phosphoric acid we use a circular bath 15 inches in diameter and 5 inches deep, with a perforated shelf about 2½ inches from the bottom, on which the flasks rest while digesting. Attached to the bath is a clockwork arrangement, which, by agitating the water with paddles, keeps it at a uniform temperature. Our flasks are small Erlenmeyers, holding about 300 cc. The temperature of the water in the bath is not sensibly lowered by the introduction of the flasks. The citrate solution in the flasks never reaches a temperature of 65° C., but after from eight to ten minutes, depending somewhat on thickness of glass of flasks, reaches between 63° and 64° where it stops.

"In filtering No. 3 I was greatly troubled by the extreme "muddiness" of the filtrate until I used a heavy packing of asbestos, by which means I got a clear filtrate. Mr. Perkins, however, who used filter paper, using as high as four papers at a time, never succeeded in getting a clear filtrate, which will possibly account for my results being higher on the citrate-insoluble in that sample.

"Two determinations of alumina in No. 1 by Mr. Wilkinson's method and one by Mr. Krug's method gave results nearly 1 per cent apart, the first being 2.63 per cent. A thorough investigation of this subject is recommended.

(9) *Agricultural Experiment Station of Illinois.*—Reported by E. H. Farrington, Champaign, Ill. Moisture determined in water oven. The total phosphoric acid was determined by H_2SO_4 and $NaNO_3$; also by $Mg(NO_3)_2$. The tabulated results are the averages, except in the case of No. 1.

Phosphoric acid by two methods.	1.	2.	3.	4.
$H_2SO_4 + NaNO_3$	17.76	13.92	21.92	18.40
$Mg(NO_3)_2$	17.73	13.95	22.14	18.82

The ammonium citrate was neutralized by "Huston's method."

(10) *New Hampshire Experiment Station.*—Reported by F. W. Morse, Hanover, N. H. Analytical work by F. W. Morse and E. P. Stone. Moisture determined in an air bath with gas regulator at 100°. Ammonium citrate neutralized by the $CaCl_2$ method.

Total in No. 1 by aqua regia; in No. 2 by $Mg(NO_3)_2$.

The bath is a circular one, of galvanized iron. It is 12 inches in diameter, 5 inches deep, and has a cover pierced with 4 holes, each 3½ inches in diameter. The bath is filled up to within half an inch of the top, and the flasks are placed in the holes and allowed to sink into the bath as far as they will. Erlenmeyer flasks of 250 cc. capacity are used. The bath is heated by a Bunsen burner, and the water is kept in

circulation by a hand blower. A thermometer fixed in a small hole in the center of the cover indicates the temperature.

The bath, when in use, contains from 7,500 cc. to 7,800 cc. Four flasks will contain 400 cc. of solution; therefore the ratio of the liquids is 1 to 19.

The bath falls barely 1° on adding the flasks, and is almost immediately brought back to 65° by increasing the heat.

Test of bath and contents of flask.

	First trial.		Second trial.	
	Bath.	Flask.	Bath.	Flask.
	°	°	°	°
5 minutes	65.5	40	65	55
10 minutes	65.0	55	65	59
15 minutes	65.0	60	65	62
20 minutes	64.5	62	65	62
25 minutes	64.5	62	66	63
30 minutes	65.0	63	66	63

(11) *Vermont Experiment Station.*—Joseph L. Hills, Burlington, Vt., analyst. In Nos. 1, 2, and 3 the addition of ammonium citrate to the wash water seemed to be without effect in time, manipulation, or result.

Bath for insoluble acid, 20 by 20 by 8 inches; flasks, 200 cc. graduated; time to attain temperature, five minutes; citrate neutralized by $CaCl_2$ and was 100.05 per cent of truth, or very faintly alkaline.

Table of comparison of methods on total.

	1.	2.	3.	4.
$Mg(NO_3)_2 + HCl$	16.25 16.31	13.01 13.10	21.02 20.90	17.56
$H_2SO_4 + KNO_3$	16.08	13.28 13.00	20.85 20.90	17.76 17.63
$H_2SO_4 + Hg$	16.23	13.25 13.25	21.02 21.06 20.70	17.78 17.86
Ignition and precipitation by molybdate, hot	16.43	.....	.....	17.81
Stood over night at room temperature, then shaken violently 3 minutes	16.35	.....	.....	17.41

Insoluble phosphoric acid.

	1.	2.	3.	4.
Water-washed	6.71	4.02	6.73	10.31
Citrate-washed	6.92	4.13	6.90	9.63

The results in Table No. I are averages of the above and also of close duplicates on water soluble.

(12) *Virginia Agricultural and Mechanical College, Blacksburg, Va.*—Reported by R. J. Davidson. Work done by Mr. Davidson and T. L. Watson. The reported results are averages of closely-agreeing duplicates, except when otherwise stated.

The bath was of copper, dimensions 14½ by 13½ by 5 inches, and the water was of

such a depth in it as to reach a little above the level of the liquid in the flasks when they were placed in it. The number of flasks put in was, in the first case, 6, and in the second, 4.

The time required for the liquid in the flasks to reach the temperature of 65° was ten minutes.

The flasks used were Erlenmeyer, of about 300 cc. capacity.

TABLE I.—*Result of comparison of methods on total and insoluble phosphoric acid.*

	No. 1.	No. 2.	No. 3.	No. 4.
Total P_2O_5	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*
25 cc. of HCl+8 cc. HNO_3	17.29	.....	.....	.....
Evaporation with $Mg(NO_3)_2$, ignition and solution in HCl	17.22	14.03	22.16	.....
De Roode's method, H_2SO_4+HgO	17.09	13.90	21.94	.....
Do	17.06	14.02	22.01	.....
De Roode's method, H_2SO_4+HgO+salicylic acid+Zn dust	17.23	14.07	21.95	.....
Do	Lost	14.13	21.99	.....
Burney's method, H_2SO_4+KNO_3	.....	14.07	.....	.....
HNO_3 in excess, 30 cc. HNO_3 with a few drops of HCl	.....	.....	22.12	18.31
HCl in excess, 20 cc. HCl+10 cc. HNO_3	.....	.....	.....	18.37
Citrate insoluble.				
After removing sol. P_2O_5, residue digested with neutral am. citrate solution, filtered washed with H_2O of about 65°	6.66	5.06	7.41	.....
After removing sol. P_2O_5, residue digested with neutral am. citrate solution, filtered washed with 25 per cent neut. am. cit. sol. of about 65°	6.89	5.15	8.85	.....
Substance treated directly with neutral cit. of am. digested, filtered, washed with water of about 65°	8.28	5.68	9.81	12.38

(13) *University of Arizona.*—Reported by Mr. Collingwood; G. J. A. Heberly, analyst. Total P_2O_5, determined by $Mg(NO_3)_2$.

(14) *New Jersey Agricultural College Experiment Station.*—Reported by Mr. C. S. Cathcart, New Brunswick, N. J. Moisture determined at 100° in an air bath. Great care was used to keep the temperature at 100°.

(15) *Ohio Experiment Station, F. J. Falkenbach, acting chemist, Columbus, Ohio.*—Total phosphoric acid by $Mg(NO_3)_2$. Moisture determined in an air bath at 100°.

(16) *Massachusetts State Experiment Station.*—Reported by Dr. C. A. Goessmann; analyst, R. B. Moore. Comparison of results of different methods on total phosphoric acid.

	1.	2.	3.
	Per ct.	*Per ct.*	*Per ct.*
$Mg(NO_3)_2$	17.42	13.75	22.72
DeRoode's method	16.66	13.89	22.77
Solution in HCl+HNO_3	17.29	13.80	22.77

All tests were made according to official methods. The digestion with ammonium citrate was conducted in a rectangular copper water bath, 10 by 8 by 6 inches. About 4.5 liters of water were put in the bath. Two 8-ounce Florence flasks were used at once. On the introduction of the flasks the water in the bath sunk about 1.5°, but came again to 65° in about two minutes. The contents of the flask came to 65° in less

than two and one-half minutes. Washing on the filter was with water at about 65. De Roode's method was followed out as though no nitrates were present. In No. 1 this method failed repeatedly to give results like those obtained by the other methods."

Mr. W. J. Williams, chemist for the Walton & Whann Company, Wilmington, Del.—"To get all the total, it is necessary to use more HCl than is commonly supposed—*e. g.*, if 30 cc. HNO_3 and a small quantity of HCl be used, the totals will be too low even in phosphate made from ordinary South Carolina rock. Where there is much Fe_2O_3 and Al_2O_3 present they will be lower still. The only safe plan is to use equal quantities, or at least a sufficiency, of HCl and HNO_3. For concentrated phosphate such as the Keystone, it is necessary to use at least 20 cc. HCl and 20 cc. HNO_3 on 2 grams.

"I always take moistures in a water oven; the temperature then can not get over 212° F. or 100°. Moreover, it is always advisable to set the watch glasses on the tray and not on the floor of the oven where water of hydration is apt to be driven off.

"If you are working specially on Keystone concentrated it may assist you to say that I have found that if kept in neutral ammonium citrate at 65° for forty-five to fifty minutes from moment of insertion, practically the whole of the 'available' goes into solution; or, in other words the insolubles are practicaliy as low as if kept at that temperature for more than fifty minutes. That is, in fifty minutes ammonium citrate at 65° has extracted practically all. At thirty minutes the solvent action is still working rapidly, and it is owing to its being incomplete that the discrepancies arise.

Fifty minutes in bath = about forty minutes at 65° practically extracts all the "reverted." A longer time gives only small additions to "reverted," or small deductions from "insoluble."

Forty minutes in bath = about thirty minutes at 65° has so far completed the extraction that the differences in different hands are not very marked but reasonable.

Thirty minutes in bath = about twenty minutes at 65° the official time is too short. Solvent action going on rapidly at end of time, consequently large discrepancies.

"If the directions for nonacidulated goods read 'keep at 65° for thirty minutes,' the discrepancies would become much smaller. If it read 'keep at 65° for forty minutes, they would be of little practical moment.

"Up to two or three years ago the directions read 'keep at 65° for thirty minutes.' This is much more reasonable than 'keep in bath for thirty minutes,' because in 'keep at 65° for thirty minutes' there is a definite time at definite temperature. 'Keep in in bath thirty minutes' gives an indefinite time at indefinite or varying temperature.

DISCUSSION OF RESULTS.

The mass of material placed in the hands of the reporter, and given in abstract in the foregoing, has been supplemented by experimental work with a view of reaching the cause of the large variations shown.

The results will be discussed under their several headings.

(1) *Water.*—The determinations of water are absolutely without value in Nos. 1 and 2; not much better in No. 3; No. 4 shows more satisfactory results.

The character of the chemists doing the work, as well as the simple nature of the determination, may be taken as a guarantee that such wide variations are not due to "carelessness." A number of analysts have given no description of the details of their determinations of moisture, but taking those who have, we can divide the work into two sets—those using an air bath and regulating the temperature by an inclosed thermometer, and those using a steam or water bath in which the temperature was regulated by boiling water surrounding the air space. The reasons for the two modes of procedure being apparently the two conditions laid down in the official method, "a steam bath" and "at 100°". The average steam bath does not regis-

ter, as some state, and as our experience has confirmed, more than 95° to 98° on an inclosed thermometer.

Dividing the results on these lines we have the figures given in Table II.

TABLE II.—*Results on moisture.*

STEAM BATH.

Analyst.	Sample 1.	Sample 2.	Sample 3.	Temperature.
	Per cent.	*Per cent.*	*Per cent.*	°
H. E. Curtis	3.41	9.67	6.66	98
R. C. Kedzie	2.76	9.43	5.90	95
L. G. Patterson	3.42	9.64	6.40	100
W. R. Perkins	3.39	9.59	6.38	100
E. H. Farrington	3.34	9.90	6.55	(?)
N. W. Lord	3.47	10.50	6.60	98
W. J. Williams	3.10	9.40	6.25	(?)
Average	3.27	9.73	6.39	

AIR BATH.

	Per cent.	*Per cent.*	*Per cent.*	°
R. C. Kedzie	6.12	14.47	7.42	102
F. W. Morse	7.27	14.72		100
C. S. Cathcart	8.73	16.05	7.57	100
F. J. Falkenbach	8.11	16.05	7.73	100
W. B. Viets	7.80	15.85	7.60	101
Average	7.61	15.43	7.58	

The steam bath results are much the more concordant. To explain this rather extraordinary fact that Sample No. 1 dried at 100° in an air bath lost an average of 7.6 per cent while at 100° to 98° in a steam bath it only lost 3.27 per cent we made the following experiments. The facts were made the more certain because one result in each set was made in our laboratory, Mr. Viets, my assistant, determining the moisture in our air bath, while I made my determinations in a steam bath. His figures closely approximate the average of the air-bath results, though they were obtained before the other reports were received. The temperature was carefully regulated and constantly read by a thermometer inclosed as usual. The air bath was double walled and heated by a gas burner underneath, while the material to be dried was set in watch crystals, on a perforated metal shelf some 4 inches from the bottom of the oven.

The temperature ranged from 100° to 102° by the inclosed thermometer.

I then made a set of moisture determinations using a double-walled water oven boiling the water hard to avoid local cooling. The temperature range was as follows, read on an inclosed thermometer placed as in the air bath:

	Degrees.
Beginning of drying	93
First half hour	99
Second half hour	99
Seventh half hour	98
End of five hours	97

The percentages of moisture obtained, are given in Table II.

Could this difference of 1 to 3 degrees account for the variation in loss between the two sets?

It then occurred to me that the metal shelf on which the watch glasses sat might be much hotter than the air in the oven. To try this I placed a small cup full of mercury on it, and stuck the bulb of the thermometer in this, the air reading all the time 100°. The temperature shown by the mercury on the shelf where the watch glass sat was 110° to 115°. Here was, of course, the explanation of the high results obtained in the air bath. The material was really much hotter (10° to 15°) than the air temperature of the oven.

We then reconstructed the air bath, substituting for the metal shelf one of thin asbestos board supported on glass rods, and made a new set of determinations as follows:

	1.	2.	3.	4.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Moisture when substance was dried on asbestos shelf at 100° in air bath for five hours	4.4	11.75	6.53	0.80

These results are much nearer the steam bath figures. Presumably the temperature reached by this material was about 103° to 104°. To further investigate the water in the samples 2 grams of each were exposed to the air of the laboratory for some time and charge of weight noted.

	Sample 1.	Sample 2.	Sample 3.	Sample 4.
Loss of weight on exposure to air—	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1 hour	.05	3.60	2.65	.20
6 hours	.20	5.30	3.35	.20
24 hours	.10	5.15	3.25	.15

"Air drying" hence reached constant weight in six hours and after that the slight gain probably shows change in air moisture.

Stating these various results we have the following table:

	Sample 1.	Sample 2.	Sample 3.
	Per cent.	*Per cent.*	*Per cent.*
Air exposure 6 hours (Lord)	0.20	5.30	3.35
Steam bath at 95 (Kedzie)	2.76	9.43	5.90
Steam bath at 98 (Curtis)	3.41	9.67	6.66
Steam bath at 99-98 (Lord)	3.47	10.50	6.60
Air bath with asbestos at 103-104 (Lord)	4.40	11.75	6.53
Air bath with asbestos thermometer at 100° (Falkenbach)	6.73	13.84	6.23
Air bath metal shelf at 110°–115° (Viets)	7.80	15.85	7.60

All of these results were supposed to be at 100°, as shown by the air temperature of the bath.

The table following, furnished by Mr. Falkenbach, shows the further effect of drying. Two grams were dried in watch crystals on an asbestos shelf. The thermometer which touched the asbestos read 100°:

	Sample 1.	Sample 2.	Sample 3.
	Per cent.	*Per cent.*	*Per cent.*
5 hours at 100°	6.08	13.46	7.77
6 hours at 100°	6.43	13.74	7.78
7 hours at 100°	7.08	14.49	7.97
8 hours at 100°	7.08	14.49	7.97

Temperature raised to 110°.

	Sample 1.	Sample 2.	Sample 3.
	Per cent.	*Per cent.*	*Per cent.*
1 hour	7.83	15.19	8.02
2 hours	8.28	16.16	8.45
3 hours	8.43	16.44	8.57
4 hours	8.60	16.56	8.67
5 hours	8.68	16.69	8.75
6 hours	8.73	16.71	8.85
10 hours (constant weight reached)	9.21	17.19	9.25

I think the above shows the causes of variation in "moisture." The results obtained are simply a function of the temperature reached by the material. All forms of directly heated metal ovens give temperatures considerably above those indicated by thermometers in the air of the oven, while water ovens never reach 100°, but are subject to less variation for a given locality.

That none of these determinations truly represent "moisture" would seem to be evident, for when the loss on drying varies from 5.90 to 7.80 on No. 1 and from 9.43 to 15.85 in No. 2 for a variation of 15° to 20° in temperature it can hardly be that any selected temperature between these two expels merely moisture and does not touch "water of hydration." Especially as the change is so regularly progressive as it appears to be.

In view of these facts it would seem that either the moisture determination be discarded or that all reference to temperature be omitted from the official directions and loss on drying be determined by heating five hours in a water oven in which the water be kept boiling the whole time when, except in cases where barometric conditions are decidedly different, fairly uniform results may be expected.

Total phosphoric acid.—The results on total phosphoric acid are unsatisfactory. This determination is the one of all others upon which agreement ought to be expected; it is, or should be, independent of method, and yet year after year our reports show large variations between chemists whose experience should be a guaranty of careful work. A considerable number of analysts have reported comparisons of results by several methods. We have made a number in our laboratory. Table III gives all of this work. Many of the reported figures are the averages of close duplicates, as will be seen by referring to the reports of the respective analysts.

I give the average result by each method, and how it compares, plus or minus, with the general average of Table No. III.

Referring to Table III, it appears that in all samples nearly half the results are within two-tenths per cent one way or the other of the average, most of the remaining results are within one-half per cent, and the small portion left may be classified as "scattering," and charged in part, at least, to inexperience with the work. Large plus variations offset large minus variations in each case, and the averages are little affected by the two or three extremes, and so all are included.

Table IV shows three remarkable results: (1) The close agreement between the results obtained with different methods by the same analyst; (2) the wide divergence between the results of different analysts without reference to method, and (3) the close agreement of the mean of all results by each method with the general mean of Table III. The conclusion from this table is certainly that there is no choice between the methods. That the De Roode method, both alone and as modified for nitrates, yields good results, and could with propriety be included among the official methods.

To investigate the causes of variation between various analysts, as these are obviously independent of method, the results were returned to the various chemists with the request that they investigate and report. While several reported revising

their results by, in some cases, from three-tenths to four-tenths per cent, but little information was furnished as to why it was done.

To try how much "variation" could be introduced I made a number of tests with results as follows, using sample No. 1, which seemed very constant in moisture on exposure to air.

Total phosphoric acid in No. 1.

	Per cent. P_2O_5.
(1) Mg nitrate method. large excess Mg mixture; let stand eighteen hours; precipitate somewhat flocculent, but white	17.48
(2) Another portion of same solution, stood eighteen hours, but filtered original ammonia solution of molybdate from a slight flocculent precipitate formed in it. $MgNH_4PO_4$ precipitate granular	17.30
(3) The flocculent precipitate filtered off showed only an unweighable trace of P_2O_5 and .02 per cent SiO_2. A fresh portion taken and treated with $Mg(NO_3)_2$. the residue after solution in HCl, was ground and extracted a second time (our usual practice); the second residue filtered off and fused with $NaNO_3$ and Na_2CO_3. In residue	0.02
(4) A fresh portion taken, treated as before, except that it was merely warmed with HCl twenty minutes. In residue	0.22
(5) Using the solution filtered from the low phosphorus residue, a careful determination was made. Adding a bare excess of Mg mixture so that the filtrate from $MgNH_4PO_4$ only showed a faint reaction for Mg after some seconds. Precipitate gave	16.81
(6) The same, but a considerable excess of Mg mixture was carefully added.	16.96

In both the latter cases the ammonia solution of the yellow precipitate stood twenty-four hours without showing any precipitate of SiO_2, Al_2O_3 or other impurity.

Determinations No. 2 and No. 6 were made with equal care and from the same bottle of fertilizer and should have been the same; they were weighed out at different times, however.

Mr. Falkenbach, at the Experiment Station laboratory, had taken for his work half of the sample bottle which my assistant, Mr. Viets, used for his work. Mr. Falkenbach's results on total were decidedly lower than Mr. Viets's. I then gave him a new set of samples, when he found the results in Table III different from those in Table I. We then opened a new bottle of No. 1. Mr. Viets found in a first determination 16.93 per cent. Then to try the effect of dilution of volume he weighed out another sample from this same bottle and found the following set of results:

	Phosphoric acid.
Mg precipitate in 100 cc	16.67
Mg precipitate in 200 cc	16.61
Mg precipitate in 400 cc	16.61

Showing no effect from dilution, but entirely different from his results on the same bottle of fertilizer weighed out at another time.

Mr. Farrington, at Champaign, Ill., reported to me first total P_2O_5 in No. 1, 17.76. I wrote him he was high, and he said he had determined it and got 17.86. At the same time he returned me half of his sample. This we analyzed and found total 16.87 and 16.80 by different methods. I sent those results to him and received the following:

"After receiving your first letter I had my assistant make two estimations of total P_2O_5 on the sample without his knowing what it was and he got 17.94 and 17.82 per cent. So in my second report to you I sent the result, 17.86 per cent. After sending you one-half my sample and getting your result on it of 16.87 and 16.80 per cent, I went to work again on what was left of the sample and made determinations, separating SiO_2 and not by different methods of solution of the sample. I got eight results for total P_2O_5 on No. 1, between 16.64 and 16.80 per cent, with an average of

16.72 per cent. I am unable to discover why my first results were 100 per cent higher than this, though I have diligently sought it."

The final result, 16.72, is the one given in Table III. These experiments seem to show that causes of large variation exist in the treatment of the sample rather than in the analytical work proper.

Several years ago I had great difficulty in getting duplicate determinations on a fertilizer consisting of ground bone and rock; it was very dry, and we traced the trouble to attempts to weigh out small quantities of this mixture. The least shaking sent the bone on top and the rock to the bottom. We found all attempts to use a spatula in weighing gave results far from the truth, and finally adopted the plan of mixing carefully all the sample on paper, then taking a spoon to lift small portions to the balance case, never shaking off any.

Mr. Viets suggested that the samples this year be so examined.

We opened a fresh lot of bottles, and made a "mechanical analysis," by putting portions of 10 grams through a fifty-mesh sieve and weighing the coarse material; then each portion was analyzed.

	Coarse material.	P_2O_5 in coarse material.	Fine material.	P_2O_5 in fine material.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
No. 1	22.5	11.95	77.5	18.15
No. 1 (duplicate)	23.3	.....	.....	.....
No. 2	30.8	15.85	69.2	12.78
No. 2 (duplicate)	30.6	.....	.....	.....
No. 3	22.7	19.62	77.3	22.24
No. 3 (duplicate)	22.2	.....	.....	.....
No. 4 (using a 90-mesh sieve)	29.0	17.57	71.0	18.40
No. 4 (duplicate)	29.9	.....	.....	.....

Further experiments were made on No. 1, using a 90-mesh sieve. Separated in this way the percentages were as follows: Coarse, 47.2 per cent $P_2O_5 = 14.19$; fine, 52.8 per cent $P_2O_5 = 19.23$, showing that the finer the sieve the richer the product. We then tested several samples of No. 1, to see if reasonable uniformity existed in the various samples.

	Coarse 90-mesh sieve (per cent.)
Last bottle opened	47.2
First bottle—Viets' sample bottle half used up	49.4
First bottle used by Lord	47.6
First bottle used by Falkenbach	47.7
Farrington's returned sample	51.3
Bottle shaken 3 minutes slightly and poured a portion out slowly	52.5
Lifted out successive portions with a spatula, as might be done in weighing: average of three results	45.6

Lastly, I add the results of the exposure of weighed amounts to the air of the laboratory, August 18, as compared with the results attained in April, when the room was heated by steam.

	1.	2.	3.	4.
Loss or gain of moisture in one hour's exposure to air of laboratory:				
April	— 0.5	— 3.60	— 2.65	— 0.20
August	+1.50	— 0.20	— 1.30	— 0.05

From these facts I submit the following conclusions: The wide variations in total P_2O_5, between different analysts are probably to be partly explained by the separation of the ingredients in the samples either by previous treatment or by want of care in weighing out, and in some cases errors occur by loss of dust (see Mr. J. S. Meng's letter); that where goods consist of mixtures of materials which have different percentages of phosphoric acid corresponding to different degrees of fineness, extreme care is necessary in mixing and weighing to get an average in so small a quantity as 2 grams, and that the fineness of the grinding prescribed by the association methods is not enough for so small amounts; that loss and gain of moisture may play a part in the variations, but it is of secondary importance.

Water-soluble phosphoric acid.—The results on this seem to be fairly satisfactory, and the only point of importance seems to be that brought out by Dr. Kedzie, that there is a continuous action as long as water is used, the limit fixed by the association being arbitrary.

Insoluble phosphoric acid.—The results are, all things considered, fairly good on No. 2 and worse on No. 4; No. 1, containing Keystone phosphate, shows great improvement over last year, when variations of 7 per cent occurred. No. 4 is in its very nature utterly unadapted to the process, and No. 3, a bone, not much better.

The extremely tedious nature of the washing of these animal phosphates is probably one of the causes of the wide divergence of results obtained.

If the question of weighing out samples plays any part in the totals it would certainly have a greater influence here, because the mechanical condition must have a large influence on the solution of the material in citrate.

Here, as in the case of the totals, the individual work of each analyst compares closely in the duplicates he reports, showing care and uniformity of work.

Table IV gives the work on insoluble phosphoric acid as far as it can be classified. For the descriptions of the various forms of water bath used reference must be made to the notes of the analyst as given in the beginning of this report.

This table shows no correspondence between conditions and results; the man who reports the shortest time for his flasks to get hot reports the highest per cent of insoluble acid. There appears to be no uniformity of result in the comparison between washing by citrate and washing with water. Our own results lead me to expect that citrate washing, especially in those fertilizers which gave so much trouble in washing, would lead to low results in insoluble; but some report higher and some lower, and some no difference.

This failure to reach any conclusion from experiments such as those tabulated is easily accounted for if the difficulty lies in the weighing out of an average 2 grams of the substance in the state of division it is brought to by the official methods of sampling.

We have tried another mode of approaching the subject of water and citrate washing, which was suggested by Mr. Huston's work last year, and which is independent of the absolute amounts present.

We made two determinations of insoluble acid in each sample, proceeding exactly by this year's directions, using 200 cc. Erlenmeyer flasks, 2 grams of substance, and strictly neutral citrate, tested by Mr. Huston's $CaCl_2$ method. The flasks were emptied into 12 cm ribbed filters and contents transferred as usual, all the liquid being allowed to run through; then the filters were washed, one with water at 65° and the other with water and 1.09 citrate solution (1:4), the filters being filled and allowed to drain six times. These six fillings were received into separate beakers and each two evaporated to dryness with $Mg(NO_3)_2$ ignited and the P_2O_5 determined, each determination being made on from 75 to 100 cc. of washings.

The following are the results:

Water wash.

	No. 1.	No. 2.	No. 3.
Per cent P_2O_5 in—			
First two washings	1.00	.54	3.29
Second two washings	0.29	.14	0.14
Third two washings	0.09	.05	0.06

These per cents are calculated on the substance used; that is, it represents the per cent of difference on the fertilizer taken.

Citrate wash (1:4).

	No. 1.	No. 2.	No. 3.
Per cent P_2O_5 in—			
First two washings	.85	.64	3.20
Second two washings	.43	.08	1.13
Third two washings	.27	.14	0.61

These results are certainly indicative of one fact that the citrate wash water on material like No. 3, which contains bone, exerts a continuous solvent action while water does not; the same solvent action appears in No. 1 and even in No. 2, though in far less degree.

To sum up then the results of the work on insoluble phosphoric acid determinations it would appear—

(1) That details of water baths and flasks fail to show any reason for variations in results.

(2) That comparison of results by different analysts as to action of water, or of citrate and water, in washing insoluble acid give entirely conflicting conclusions.

(3) That my own direct experiment shows progressive solvent action of the citrate wash, while water action soon ceases in all three cases.

In suggesting that a probable cause for variation is the lack of uniformity in the 2 grams weighed out for treatment, it is evident that lack of uniformity in the samples sent out by the reporter would equally explain all the differences. It is necessary to consider the probability of this.

The samples sent out were large (3-ounce bottles full). All had been put through a 25-mesh sieve. They were carefully mixed several times and then put into the bottles immediately, and small portions into each bottle, not filling the bottles consecutively but simultaneously; furthermore, various bottles opened here have shown a practically identical mechanical analysis, and when a number of results were obtained a practically identical analysis. Correspondence with Mr. Farrington shows the variations that were obtained from one bottle of material. My own varying results were also from a single bottle.

That the ingredients may easily have become separated in transit from shaking is probable, and failure to carefully remix would of course account for the trouble, supposing the material used direct from the bottle.

As a further evidence of the uniformity of the samples as sent out, we took all the bottles we had on hand after concluding our work and weighed 10 grams from each, made the mechanical analysis, and then dissolved the 10 grams in HCl, using 100cc., treating with $KClO_3$, and after thorough boiling diluting to 500cc. in 25cc. of each solution. Thus the P_2O_5 was determined.

The following were the results obtained:

	Coarse material.	P_2O_5.
	Per cent.	*Per cent.*
(1) First bottle opened in March, only a little left	24.8	16.43
(2) Bottle opened in April used in my tests	23.9	16.45
(3) Mr. Farrington's returned sample, a small amount	25.8	16.43
(4) New bottle given to Mr. Falkenbach, nearly full	23.8	16.76
(5) Bottle that was opened in July	23.7	16.58
(6) Bottle that was opened in August	22.3	16.81
(7) New bottle especially opened for test	23.5	16.77

With the exception of the first three results the results are practically identical. The $KClO_3$+HCl solution was probably not perfect on so large an amount, but the agreement of results is such as seems to establish identity of samples. The first three results represent goods opened a long time.

Determinations of alumina which were asked for were only made by one or two chemists; the results were so extremely divergent that they are not given. It would be very desirable that the subject of the determination of alumina in fertilizers be investigated by the reporter for the coming year.

TABLE III.—*Results reported as official.*

SAMPLE No. 1.

Analyst.	Water.	Phosphoric acid.						
		Water soluble.	Citrate soluble.	Available.	Insoluble.	Difference from average insoluble.	Total.	Difference from average total.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
U. S. Dept. Agriculture—K. P. McElroy, Washington, D. C	7.12	2.06	7.29	9.35	7.51	— .41	16.86	—.26
Pa. Ag. Ex. Station—A. W. Fries, State College, Pa.	3.84	2.11	7.58	9.69	7.94	+ .02	17.63	+.51
Kentucky Ag. Ex. Station—H. E. Curtis, Lexington, Ky	3.41	1.67	6.89	8.56	9.09	+1.17	17.65	+.53
Alabama State Laboratory—R. E. Noble, Auburn, Ala	8.79	1.70	8.01	9.71	7.41	— .51	17.12	.....
Clemson Ag. Col.—F. S. Shiver, Fort Hill, S. C	3.78	.....	.....	.....	.....	.....	17.24	+.12
Clemson Ag. College—R. N. Brackett	.....	.....	.....	.....	.....	.....	17.21	+.09
North Carolina Ag. Ex. Station—J. S. Meng, Raleigh, N. C.	7.64	1.94	7.86	9.80	8.42	+ .56	17.22	+.10
North Carolina Ag. Ex. Station—F. B. Carpenter	.....	1.93	7.13	9.06	8.13	+ .21	17.19	+.07
Michigan Ag. Col.—R. C. Kedzie, Ingraham Co., Mich	2.76 / 6.12	1.90	7.56	9.46	7.48	— .44	16.94	—.18
Mississippi Ag. Ex. Station—L. G. Patterson, Starkville, Miss	3.42	1.79	7.48	9.27	8.26	+ .34	17.53	+.41
Mississipi Ag. Ex. Station—W. R. Perkins	3.39	1.73	7.47	9.20	8.35	+ .43	17.55	+.43
Ag. Ex. Station—E. H. Farrington Champaign, Ills	3.34	1.92	7.57	9.49	7.23	— .69	16.72	—.40
New Hampshire Ex. Station—F. W. Morse and E. P. Stone, Hanover N. H.	7.27	2.03	6.58	8.61	8.27	+ .35	16.88	—.24

TABLE III.—*Results reported as official*—Continued.

SAMPLE No. 1—Continued.

Analyst.	Water.	Phosphoric acid.						
		Water soluble.	Citrate soluble.	Available.	Insoluble.	Difference from average insoluble.	Total.	Difference from average total.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Vermont Ag. Ex. Station—J. L. Hills, Burlington, Vt	3.36	1.74	7.72	9.46	6.81	—1.11	16.27	—.85
Virginia Ag. and Mech. Col.—T. L. Watson and R. J. Davidson, Blacksburg, Va	3.34	1.89	8.65	10.54	6.66	—1.26	17.20	+.08
Arizona Ex. Station—J. A. Heberly, Tucson, Ariz.*	13.06	2.13	6.21	8.34	8.42	+ .50	16.76	—.36
New Jersey Ag. Col. Ex. Station—C. S. Cathcart, New Brunswick, N. J.	8.73	1.99	5.57	7.56	8.93	+1.01	16.49	—.63
Ohio State Ex. Station—F. J. Falkenbach, Columbus, O	8.11	1.68	8.55	10.23	6.64	—1.28	16.87	—.25
Ohio State University—N. W. Lord, Columbus, O.†	3.47	1.68	7.60	9.28	7.86	— .06	17.14	+.02
Ohio State University—W. B. Viets‡	7.80	1.92	7.41	9.33	7.67	— .25	17.00	—.12
Massachusetts Ag. Ex. Station—R. B. Moore, Amherst, Mass	8.42	2.39	5.82	8.21	9.21	+1.29	17.42	+.30
Baugh & Sons Co.—F. Bergami, Philadelphia, Pa	3.58	2.08	6.88	8.96	8.32	+ .40	17.28	+.16
Walton & Whann Co.—W. J. Williams, Wilmington, Del	3.10	2.11	7.94	10.05	7.68	— .24	17.73	+.61
Average of all results		1.92	7.32	9.24	7.92		17.12	
Per cent of results:								
Within 0.2 per cent of average		71.40				9.50		43.50
Within 0.4 per cent of average		95.20				38.00		69.60
Within 0.7 per cent of average		All				71.40		95.70
Over 1 per cent from average						26.10		None
Extreme difference between highest and lowest		0.72				2.57		1.46

SAMPLE No. 2.

U. S. Dept. Agriculture—K. P. McElroy, Washington, D. C	13.99	2.73	6.59	9.32	4.55	— .61	13.87	—.08
Pa. Ag. Ex. Station—A. W. Fries, State College, Pa	11.00	2.81	6.10	8.91	5.52	+ .36	14.43	+.48
Kentucky Ag. Ex. Station—H. E. Curtis, Lexington, Ky	9.67	2.14	6.36	8.50	5.80	+ .64	14.30	+.35
Alabama State Laboratory, R. E. Noble, Auburn, Ala	16.69	2.41	6.60	9.01	5.39	+ .23	14.40	+.45
Clemson Ag. Col.—M. B. Hardin, F. S. Shiver, Fort Hill, S. C	10.05						13.91	—.04
Clemson Ag. Col.—R. N. Brackett							13.93	— 02

*Moisture at 130° C., except lower results at 100°.

†Dried in steam bath, thermometer 97°–98°.

‡Dried in air bath; temperature indicated by suspended thermometer, 101°.

TABLE III.—*Results reported as official*—Continued.

SAMPLE No. 2—Continued.

Analyst.	Water.	Phosphoric acid.						
		Water soluble.	Citrate soluble.	Available.	Insoluble.	Difference from average insoluble.	Total.	Difference from average total.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
North Carolina Ag. Ex. Station—J. S. Meng, Raleigh, N.C	15.55	2.55	6.19	8.74	5.05	— .11	13.79	—.16
North Carolina Ag. Ex. Station—F. B. Carpenter	.....	2.63	5.89	8.52	5.26	+ .10	13.78	—.17
Michigan Ag. Col.—R. C. Kedzie, Ingraham Co., Mich	{ 9.43, 14.47 }	2.55	6.08	8.63	5.18	+ .02	13.81	—.14
Mississippi Ag. Ex. Station—L. G. Patterson, Starkville, Miss	9.64	2.25	6.28	8.53	5.67	+ .51	14.20	+.25
Mississippi Ag. Ex. Station—W. R. Perkins	9.59	2.28	6.14	8.42	5.73	+ .57	14.15	+.20
Ag. Ex. Station—E. H. Farrington, Champaign, Ills	9.90	2.56	6.49	9.05	4.89	— .27	13.94	—.01
New Hampshire Ex. Station—F. W. Morse and E. P. Stone, Hanover, N. H	14.72	2.92	5.53	8.45	5.60	+ .44	14.05	+.10
Vermont Ag. Ex. Station—J. L. Hills, Burlington, Vt	10.76	2.31	6.76	9.07	4.08	—1.08	13.15	—.80
Virginia Ag. and Mech. College, T. L. Watson and R. J. Davidson, Blacksburg, Va	9.69	2.59	6.39	8.98	5.06	— .10	14.04	+.09
Arizona Ex. Station—J. A. Heberly, Tuscon, Ariz.*	20.73	2.80	5.23	8.03	5.57	+ .41	13.60	—.35
New Jersey Ag. Col. Ex. Station—C. S. Cathcart, New Brunswick, N. J	16.05	2.76	5.46	8.22	5.36	+ .20	13.58	—.37
Ohio State Ex. Station—F. J. Falkenbach, Columbus, O	16.05	2.42	8.16	10.58	3.37	—1.79	13.95	.....
Ohio State University—N. W. Lord, Columbus, O.†	10.50	2.27	6.59	8.86	5.16	.....	14.02	+.07
Ohio State University—W. B. Viets‡	15.85	2.68	6.46	9.14	4.79	-- .37	13.93	—.02
Massachusetts Ag. Ex. Station—R. B. Moore, Amherst, Mass	14.92	3.10	5.35	8.45	5.30	+ .14	13.75	—.20
Baugh & Sons Co.—F. Bergami, Philadelphia, Pa	10.00	2.56	5.81	8.37	5.63	+ .47	14.00	+.05
Walton & Whann Co.—W. J. Williams, Wilmington, Del	9.40	2.75	6.15	8.90	5.44	+ .28	14.34	+.39
Average of all results	.....	2.57	6.22	8.79	5.16	.....	13.95	.....
Per cent of results:								
Within 0.2 per cent of average	.....	57.00	.....	.....	.....	33.30	.....	65.30
Within 0.4 per cent of average	.....	90.50	.....	.....	.....	57.10	.....	87.00
Within 0.7 per cent of average	.....	All	.....	.....	.....	90.50	.....	95.70
Over 1 per cent from average	.....	.....	.....	.....	.....	9.50	.....	None
Extreme difference between highest and lowest	.....	0.96	.....	.....	.....	2.43	.....	1.28

*Moisture at 130° C., except lower results at 100°.

†Dried in steam bath, thermometer 97°–98°.

‡Dried in air bath; temperature indicated by suspended thermometer, 101°.

TABLE III.—*Results reported as official*—Continued.

SAMPLE No. 3.

Analyst.	Water.	Phosphoric acid.						
		Water soluble.	Citrate soluble.	Available.	Insoluble.	Difference from average insoluble.	Total.	Difference from average total.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
U. S. Dept. Agriculture K. P. McElroy, Washington, D. C	6.90	2.44	14.59	17.03	4.86	—3.15	21.81	— .13
Pa. Ag. Ex. Station—A. W. Fries, State College, Pa	6.43	2.61	8.86	11.47	11.15	+3.14	22.62	+ .68
Kentucky Ag. Ex. Station—H. E. Curtis, Lexington, Ky	6.66	2.09	11.20	13.29	8.95	+ .94	22.24	+ .30
Alabama State Laboratory—R. E. Noble, Auburn, Ala	8.08	2.26	10.68	12.94	9.50	+ 1.49	22.44	+ .50
Clemson Ag. Col.—F. S. Shiver, Fort Hill, S. C	6.67	.....	.....	.....	.....	.....	21.76	— .18
Clemson Ag. Col.—R. N. Brackett, Fort Hill, S. C	.....	.....	.....	.....	.....	.....	21.56	— .38
North Carolina Ag. Ex. Station—J. S. Meng, Raleigh, N. C	9.07	2.56	9.54	12.10	10.00	+1.99	22.10	+ .16
North Carolina Ag. Ex. Station—F. B. Carpenter	.....	2.46	10.31	12.77	9.34	+1.33	22.11	+ .17
Michigan Ag. Col.—R. C. Kedzie, Ingraham Co., Mich	5.90 7.42	2.52	10.17	12.69	8.80	+.79	21.49	— .45
Mississippi Ag. Ex. Station—L. G. Patterson, Starkville, Miss	6.40	2.41	14.28	16.69	5.24	—2.77	21.93	— .01
Mississippi Ag. Ex. Station—W. R. Perkins	6.38	2.31	15.01	17.32	4.55	—3.46	21.87	— .07
Ag. Ex. Station—E. H. Farrington, Champaign, Ills	6.55	2.56	13.33	15.89	6.14	—1.87	22.03	+ .09
New Hampshire Ex. Station—F. W. Morse and E. P. Stone, Hanover, H. H	.....	.....	.....	.....	.....	.....	.....	.....
Vermont Ag. Ex. Station—J. L. Hills, Burlington, Vt	6.55	2.07	12.03	14.10	6.82	—1.19	20.92	—1.02
Virginia Ag. and Mech. Col.—T. L. Watson and R. J. Davidson, Blacksburg, Va	6.40 10.53	2.49	12.16	14.65	7.41	— .60	22.06	+ .12
Arizona Ex. Station—J. A. Heberly, Tuscon, Ariz.*	7.01	2.45	9.31	11.76	10.39	+2.38	22.15	+ .21
New Jersey Ag. Col. Ex. Station—C. S. Cathcart, New Brunswick, N. J	7.57	2.46	.....	.....	.....	.....	21.22	— .72
Ohio State Ex. Station—F. J. Falkenbach, Columbus, O	7.73	2.23	10.94	13.17	8.14	+ .13	21.31	— .63
Ohio State University—N. W. Lord, Columbus, O.†	6.60	2.24	12.28	14.52	7.17	— .84	21.69	— .25
Ohio State University—W. B. Viets.‡	7.60	2.30	10.80	13.10	8.69	+ .68	21.79	— .15
Massachusetts Ag. Ex. Station—R. B. Moore, Amherst, Mass	7.46	1.75	13.04	14.79	7.93	— .08	22.72	+ .78

*Moisture at 130° C., except lower results at 100°.

†Dried in steam bath, thermometer 97°–98°.

‡Dried in air bath; temperature indicated by suspended thermometer, 101°.

TABLE III.—*Results reported as official*—Continued.

SAMPLE No. 3—Continued.

Analyst.	Water.	Phosphoric acid.						
		Water soluble.	Citrate soluble.	Available.	Insoluble.	Difference from average insoluble.	Total.	Difference from average total.
Baugh & Sons Co.—F. Bergami, Philadelphia, Pa	*Per ct.* 6.53	*Per ct.* 2.36	*Per ct.* 9.68	*Per ct.* 12.04	*Per ct.* 10.36	*Per ct.* +2.35	*Per ct.* 22.40	*Per ct.* + .46
Walton & Whann Co.—W. J. Williams, Wilmington, Del	6.25	2.56	13.31	15.87	6.72	—1.29	22.59	+ .65
Average of all results	.....	2.39	11.66	14.01	8.01	.....	21.94	.....
Per cent of results:								
Within 0.2 per cent of average	.....	80.00	.....	.....	.....	10.50	.....	40.90
Within 0.4 per cent of average	.....	95.00	.....	.....	.....	10.50	.....	59.10
Within 0.7 per cent of average	.....	All ...	.....	.....	.....	21.00	.....	86.40
Over 1 per cent from average	.....	.....	.....	.....	.....	63.20	.....	4.50
Extreme difference between highest and lowest	.....	0.54	.....	.....	.....	5.84	.....	1.80

SAMPLE No. 4.

U. S. Dept. Agriculture—K. P. McElroy, Washington, D. C	0.95	.....	9.96	9.96	8.54	—2.99	18.50	+.10
Pa. Ag. Ex. Station—A. W. Fries. State College, Pa	0.89	.....	6.63	6.63	11.98	+ .45	18.61	+.21
Kentucky Ag. Ex. Station—H. E. Curtis, Lexington, Ky	0.94	.....	6.34	6.34	12.25	+ .72	18.59	+.19
Alabama State Laboratory—R. E. Noble, Auburn, Ala	1.46	0.07	8.20	8.27	10.27	—1.26	18.54	+.14
Clemson Ag. Col.—F. S. Shiver. Fort Hill, S. C	0.99	.....	.....	.....	.....	.....	18.17	—.23
Clemson Ag. Col.—R. M. Brackett, Fort Hill, S. C	.....	.....	.....	.....	.....	.....	18.20	—.20
North Carolina Ag. Ex. Station—J. S. Meng, Raleigh, N. C	1.20	.....	4.94	4.94	13.28	+1.75	18.22	—.18
North Carolina Ag. Ex. Station—F. B. Carpenter	.....	.....	5.45	5.45	12.81	+1.28	18.26	—.14
Michigan Ag. Col.—R. C. Kedzie, Ingraham Co., Mich	0.83	.....	5.73	5.73	12.44	+ .91	18.17	—.23
Mississippi Ag. Ex. Station—L. G. Patterson, Starkville, Miss	0.99	.....	7.43	7.43	10.94	— .59	18.37	—.03
Mississippi Ag. Ex. Station—W. R. Perkins	0.89	.....	7.30	7.30	11.07	— .46	18.37	—.03
Ag. Ex. Station—E. H. Farrington, Champaign, Ills	0.75	.....	7.90	7.90	10.71	— .82	18.61	+.21
New Hampshire Ex. Station—F. W. Morse and E. P. Stone, Hanover, N. H	.....	.....	.....	.....	.....	.....	.....	.....
Vermont Ag. Ex. Station—J. L. Hills, Burlington, Vt	0.97	0.03	7.69	7.72	9.97	—1.56	17.69	—.71
Virginia Ag. and Mech. Col.—T. L. Watson and R. J. Davidson, Blacksburg, Va	1.12 1.54	.....	5.96	5.96	12.38	+ .85	18.34	—.06

TABLE III.—*Results reported as official*—Continued.

SAMPLE No. 4—Continued.

Analyst.	Water.	Phosphoric acid.						
		Water soluble.	Citrate soluble.	Available.	Insoluble.	Difference from average insoluble.	Total.	Difference from average total.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Arizona Ex. Station—J. A. Heberly, Tuscon, Ariz.*	1.11	.....	.....	.....	.....	.....	18.21	—.19
New Jersey Ag. Col. Ex. Station—C. S. Catheart, New Brunswick, N. J.	1.19	.....	5.24	5.24	12.75	+1.22	17.99	—.41
Ohio State Ex. Station—F. J. Falkenbach, Columbus, O	1.21	.....	.....	.....	.....	.....	18.62	+.22
Ohio State University—N. W. Lord, Columbus, O.†	.93	.....	7.49	7.49	11.09	— .44	18.58	+.18
Ohio State University—W. B. Viets‡	1.22	.....	5.37	5.37	13.10	+1.57	18.47	+.07
Massachusetts Ag. Ex. Station—R. B. Moore, Amherst, Mass	1.18	.....	7.01	7.01	12.13	+ .60	19.14	+.74
Baugh & Sons Co.—F. Bergami, Philadelphia, Pa	.....	.....	.....	.....	.....	.....	.....	.....
Walton & Whann Co.—W. J. Willirms, Wilmington, Del	0.75	0.26	8.13	8.39	10.43	—1.10	18.82	+.42
Average of all results	.....	.....	6.87	6.89	11.53	.....	18.40	.....
Per cent of results:								
Within 0.2 per cent of average	.....	.....	.....	.....	.....	.....	.....	57.10
Within 0.4 per cent of average	.....	.....	.....	.....	.....	.....	.....	80.90
Within 0.7 per cent of average	.....	.....	.....	.....	.....	.....	.....	95.20
Over 1 per cent from average	.....	.....	.....	.....	.....	.....	.....	None
Extreme difference between highest and lowest	.....	.....	.....	.....	.....	.....	.....	1.45

* Moisture at 130° C., except lower results at 100°.

† Dried in steam bath, thermometer 97°–98°.

‡ Dried in air bath; temperature indicated by suspended thermometer, 101°.

TABLE IV.—*Comparative results on total phosphoric acid by different methods.*

Analyst.	$H_2SO_4 + KNO_3$ or $NaNO_3$ (Barney's method).			Solution in $HCl + HNO_3$ or $KClO_3$ or HNO_3 + little HCl.			$H_2SO_4 + HgO$ or Hg (De Roode's method).			$H_2SO_4 + HgO$ with zinc and salicylic acid or salicylic acid and sodium thiosulphate.			Solution in HCl after ignition with $Mg(NO_3)_2$.		
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
K. P. McElroy	16.86	13.87	21.81												
A. W. Fries				17.63	14.43	22.62									
H. E. Curtis				17.63			17.65	14.30	22.24	17.73	14.28	22.16			
R. E. Noble													17.12	14.40	22.44
F. S. Shiver	17.24	13.91	21.76	17.21		21.75								13.74	
R. N. Brackett	17.21	13.93	21.56	16.98	13.64	21.62									
J. S. Meng				17.22	13.79	22.10									
F. B. Carpenter				17.19	13.78	22.11									
R. C. Kedzie							16.73	13.85	21.51	16.79	13.75	21.43			
E. H. Farrington	17.76	13.92	21.92										17.73	13.95	22.14
F. W. Morse				16.88										14.05	
J. L. Hills	16.08	13.14	20.88				16.23	13.25	20.89				16.28	13.06	20.96
Davidson & Watson		14.07		17.29		22.12	17.07	13.96	21.97	17.23	14.10	21.97	17.22	14.03	22.16
J. A. Heberly													16.76	13.60	22.15
F. J. Falkenbach							17.22	13.77	21.50				16.98	14.09	21.62
R. B. Moore				17.29	13.80	22.77	16.66	13.89	22.77				17.42	13.75	22.72
W. J. Williams				17.73	14.34	22.59									
W. B. Viets				16.81	13.80	21.66	16.94	13.99	22.05				17.00	13.93	21.79
Average of all	17.05	13.81	21.59	17.26	13.94	22.15	16.93	13.85	21.85	17.25	14.04	21.85	17.06	13.86	22.00
Variation from general average	—.07	—.14	—.35	+.14	—.01	+.21	—.19	—.10	—.09	+.13	+.09	—.09	—.06	—.09	+.06

TABLE V.—*Work on insoluble phosphoric acid.*

Analyst.	Insoluble residue washed with water at 65°–70°.				Insoluble residue washed with water+am. citrate.				Kind of flask used in digestion.	Capacity of flasks.	Capacity of water bath used.	Time required for contents of flask to reach maximum temperature.	Maximum temperature attained by contents of flask.	Method of testing neutrality of am. cit. solution.	Remarks.
	1	2	3	4	1	2	3	4							
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*		*cc.*	*Cu. in.*	*Min.*	°		
A. W. Fries	7.94	5.52	11.15	11.98	8.04	5.52	11.09	11.96	Erlenmeyer.	300	2,000	14	63	Corallin	Water+$\frac{1}{10}$ citrate bath dropped $\frac{1}{2}$° on adding flask.
H. E. Curtis	.....	.....	.....	.....	9.09	5.80	8.95	12.25	....do	250	1,000	12	63	Litmus $CaCl_2$	Water+$\frac{1}{3}$ citrate.
Meng & Carpenter	.....	.....	.....	.....	.....	.....	.....	.....	....do	200	1,600	15	$64\frac{1}{2}$	.....	
R. C. Kedzie	7.57	5.24	.....	12.60	7.42	5.11	.....	12.28	.....	.....	.....	$3\frac{1}{2}$	.....	.....	Water+25 per cent citrate; introduction of flask lowered temp. of bath 1°.
Patterson & Perkins	.....	.....	.....	.....	.....	.....	.....	.....	Erlenmeyer.	300	900	8–10	64	.....	
E. H. Farrington	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	$CaCl_2$	
F. W. Morse	.....	.....	.....	.....	.....	.....	.....	.....	Erlenmeyer.	250	600	15	62–63	$CaCl_2$	
J. L. Hills	6.71	4.02	6.73	10.31	6.92	4.13	6.90	9.63	Graduated flasks.	200	3,200	5	.....	$CaCl_2$	
Davidson & Watson	6.66	5.06	7.41	.....	6.89	5.15	8.85	.....	Erlenmeyer.	300	900	10	.....	.....	
Do	8.25	5.63	9.81	12.38	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	Treated without previous water extraction.
R. B. Moore	9.21	5.36	7.93	12.13	.....	.....	.....	.....	.....	250	500	2	.....	.....	
Lord & Viets	7.60	5.01	8.50	12.52	.....	.....	.....	.....	Erlenmeyer.	200	600	10	65	$CaCl_2$ and cochineal.	Rubbed up before adding citrate; washed with cold water.
Do	7.67	4.79	8.63	13.10	7.54	4.85	6.77	'11.12	....do	200	.....	10	65	.....	
N.W. Lord	7.86	5.16	7.17	11.09	.....	.....	.....	.....	....do	200	.....	10	65	.....	

At the conclusion of Mr. Lord's report, papers on subjects relative thereto were called for. Mr. Huston presented the following paper:

DETERMINATIONS OF LOSS ON HEATING SAMPLES OF PHOSPHATES SENT OUT BY THE REPORTER ON PHOSPHORIC ACID FOR 1892.

By H. A. Huston.

The researches of Stocklasa have shown that pure superphosphates decompose at temperatures much exceeding 105°. In the case of such goods as the fertilizer chemist is obliged to deal with, the conditions are more complicated, as there is water present as moisture, water of crystallization, and water of constitution. The accompanying tables give results at various temperatures other than the official. When the temperature of 135° was used partial decomposition of the superphosphates had doubtless taken place. The use of a current of dry gas increases the amount of loss very considerably, even in the case of the basic slag. In case of basic slag a gain in weight is noted after heating for some hours, a result that is similar to those reported by Mr. Knorr and myself last year on bone and on phosphate of alumina.

In a number of cases it is noted that the material increases in weight or that the weight remains constant for ten hours, but that on further heating the weight again decreases. So that at the temperatures employed it seems to be impracticable to obtain a constant weight within any such time as could reasonably be devoted to drying samples in current work.

In view of the wide divergence in the results of different analysts and the great differences in duplicate determinations in the hands of the same analysts, it is suggested that it would be wise to abandon the determination of water in samples when possible, and that in cases where such determinations must be made that the results reported be not called simply water, but rather the amount of loss on heating for five hours at 100° in a water oven with the water in brisk ebullition.

Dried at 100°, boiling water jacket, official method.

	Series 1.				Series 2.			
	1	2	3	4	1	2	3	4
5 hours	3.460	10.530	6.560	0.820	2.795	10.550	6.200	0.83
10 hours	3.259	11.875	6.250	0.865	2.915	12.150	5.950	0.73
15 hours	3.460	12.700	6.460	0.875	3.600	12.525	6.375	0.77
20 hours	3.665	12.935	6.095	0.785	3.990	12.825	6.375	0.84
25 hours					4.190	13.000	6.775	
30 hours					3.950	13.160	6.850	
40 hours					4.675	14.000	6.950	
50 hours					4.850	14.000	7.160	
60 hours					5.050	14.275	7.100	
70 hours					5.250	14.350	7.335	
80 hours					5.400	14.335	7.385	

Water in current of dry hydrogen at 100°.

[Tubes used same as for fodder analyses.]

	No. 1.		No. 2.		No. 3.		No. 4.	
	A.	*B.*	*A.*	*B.*	*A.*	*B.*	*A.*	*B.*
5 hours	4.18	3.73	10.330	11.035	6.589	6.445	1.350	1.375
10 hours		4.96	11.765	12.850	6.915	6.110	1.375	1.340
15 hours		5.25	12.685	13.090	7.330	6.990	1.200	1.205
19 hours		5.76	13.495	13.625	7.495	7.150	1.260	1.205
23 hours		6.33			7.390	7.035	1.000	0.835

In toluol bath, 107°.

	No. 1.		No. 2.	
	A.	*B.*	*A.*	*B.*
5 hours	4.260	4.015	12.320	13.040
10 hours	4.950	4.790	13.480	13.835
15 hours	5.375	5.580	13.880	14.075
20 hours	5.955	6.080	14.180	14.400
25 hours	6.175	6.270	14.280	14.500
35 hours	6.500	6.570	14.500	14.695
45 hours	6.585	6.630	14.575	14.695
55 hours	6.790	6.770	14.640	14.770
65 hours	6.990	6.950	14.690	14.930
75 hours	7.150	7.090	14.855	15.010

In air bath, 135°.

	No. 1.		No. 2.		No. 3.		No. 4.	
	A.	*B.*	*A.*	*B.*	*A.*	*B.*	*A.*	*B.*
5 hours, series 1	7.940	8.065	15.745	15.945	8.410	7.800	.895	.865
5 hours, series 2	7.825	8.420	15.625	15.355	7.925	7.765	.920	.865

In xylol bath at 135–137°.

	No. 1.		No. 2.		No. 3.		No. 4.	
	A.	*B.*	*A.*	*B.*	*A.*	*B.*	*A.*	*B.*
5 hours	8.265	8.27	16.225	15.945	8.310	8.315	.940	1.060
10 hours	8.770	8.80	16.375	16.230	8.725	8.705	.905	1.035

Mr. De Roode. I have here a paper with some data comparing the results with the different methods.

In the first comparison, in the column marked Kjeldahl, are the percentages of P_2O_5 obtained in sample used, also for nitrogen determinations. In the second column are the percentages obtained by the official method.

In the second comparison are the data of the Gunning method as compared with the official method.

Comparison of De Roode's and official methods for P_2O_5.

Kjeldahl.	HNO_3+ HCl.	Kjeldahl.	HNO_3+ HCl.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
8.51	8.49	11.82	11.88
9.21	9.29	12.18	12.05
9.56	9.42	12.18	12.13
9.59	9.59	12.24	12.28
9.70	9.72	12.36	12.40
9.89	9.85	12.37	12.31
9.90	9.84	12.38	12.37
10.30	10.30	12.63	12.68
10.45	10.29	12.82	12.73
10.45	10.38	13.34	13.24
10.50	10.41	13.56	13.59
10.55	10.38	13.56	13.64
10.68	10.57	13.57	13.53
10.69	10.63	13.64	13.46
10.81	10.82	14.93	14.89
10.93	10.98	15.42	15.21
10.95	10.84	15.43	15.40
11.12	11.16	17.46	17.26
11.30	11.26	18.52	18.45
11.35	11.18	18.86	18.88
11.46	11.46	21.47	21.36
11.55	11.64	21.48	21.70
11.63	11.67	22.32	22.49
11.64	11.54	24.59	24.74
11.67	11.62	25.10	25.04
11.72	11.63	25.56	25.59

Comparison of the Gunning with the official method for P_2O_5.

Gunning.	HNO_3+HCl.	Gunning.	HNO_3+HCl.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
10.86	10.78	14.02	14.10
11.13	11.14	14.21	14.25
12.01	12.09	14.47	14.49
12.05	12.11	15.02	15.06
12.55	12.61	18.14	18.09
13.05	13.01	20.53	20.42
13.71	13.84		

Mr. LORD. I have a communication from Mr. Hardin, of South Carolina, "On the occurrence of metaphosphoric and pyrophosphoric acids in cotton-seed meal."

THE OCCURRENCE OF METAPHOSPHORIC AND PYROPHOSPHORIC ACIDS IN COTTON-SEED MEAL.

By M. B. HARDIN.

In the course of some experiments commenced in January last on cotton-seed meal, the determination of the water and soluble phosphoric acid was included. Instructions were given to evaporate the aqueous solutions to dryness and to ignite the residues gently before attempting to determine the phosphoric acid. At the same time, in order to see what proportion of the phosphoric acid could be obtained by direct precipitation with molybdate from the aqueous solution, Messrs. Sims and Brackett were requested to make some experiments in this direction. The results were all of the same general character, so that the following examples will be sufficient for purposes of illustration:

Analyst.	Station No.	When received.	Color of meal.	Evaporation—ignition $HCl+HNO_3$.	Dried precipitation 1 hour at 65°.
				Per cent.	*Per cent.*
R. N. Brackett	102	1891	Brownish	1.67	0.19
Do	104	1891	Dark brown	2.07	0.35
C. W. Sims	104	1891	do	1.97	0.35
R. M. Brackett	107	1891	Brownish	1.69	0.14
Do	532	1892	Dull yellow, undecorticated.	1.40	1.19
Do	408	1892	Bright yellow	1.64	0.14
C. W. Sims	408	1892	Bright yellow	1.62	0.17

In all the cases of direct precipitation, if the filtrates from the phosphomolybdate precipitates were allowed to stand at 60° additional precipitation occurred, the quantity of phosphoric acid thrown down increasing with the time of standing, but it was shown by Dr. Brackett that complete precipitation could not be effected in this way, at least in any reasonable time. Disregarding all observations and inferences not bearing upon the object of this paper, it will simply be stated that the point which attracted most attention was the singularly close agreement which, as a rule, existed between the quantities of phosphoric acid obtained from the different samples by direct precipitation. It had been expected that the organic matter would prevent, or at least interfere with the precipitation by molybdic solution, but it was difficult to see why this interference should have always about the same limit, and that nearly the same quantity of phosphoric acid should in every case escape its influence.

I next submitted a sample of meal—No. 408—to dialysis, the water in the outer cell of dialyser being changed every twelve hours. The diffusates remained clear for about eight or ten hours, but after that time became turbid, even when transferred to another vessel. At the expiration of a week the phosphoric acid which had passed through the dialyser was 1.98 per cent. Another sample of the same meal dialysed for one week in presence of phenol gave 1.83 per cent. In both of these cases the diffusates were evaporated, and the ignited residues taken up with hydrochloric acid which was then displaced by HNO_3. A third sample of the same meal was dialysed for three days. The diffusate, divided into two equal parts, yielded 0.192 phosphoric acid by direct precipitation and 0.847 by previous evaporation and

ignition. It was thus shown that all the phosphoric acid found in the turbid aqueous solution of cotton-seed meal obtained by ordinary filtration was actually in solution, or capable of becoming so, on standing; that by long contact with water some of the "citrate soluble" became soluble in water; and finally, that the causes which prevented the precipitation of the whole of the phosphoric acid from the ordinary aqueous solution were not removed by dialysis. Therefore, either the precipitation of the phosphoric acid is prevented by the small proportion of the organic matter capable of passing through a colloid septum, or the phosphoric acid itself is not all in the tribasic state. Mr. F. S. Shiver and Dr. Brackett were then requested to analyze a mixture of equal weights of an acid phosphate containing 9.27 per cent of soluble phosphoric acid and of a cotton-seed meal yielding 0.26 per cent of soluble phosphoric acid by direct precipitation, and 1.65 per cent by previous evaporation and ignition. If the organic matter of the meal does not interfere with the precipitation of the soluble phosphoric acid of the acid phosphate, the mixture ought to give 4.77 per cent of soluble phosphoric acid by direct precipitation, and 5.46 per cent by previous evaporation and ignition.

Following are the results obtained:

	Direct precipitation.	Evaporation and ignition.
	Per cent.	*Per cent.*
F. S. Shiver	4.80	5.25
R. N. Brackett	4.82	5.39

Hence the precipitation of the soluble phosphoric acid of the acid phosphate was not interfered with by the organic matter of the meal. Whether the differences between the actual and calculated results have any special significance or not can be determined only by further experiments. Considering all the work done up to this point, it seemed improbable that the deportment of the aqueous solution of a cotton-seed meal with molybdic solution could be due to the organic matter of the meal. It seemed much more probable that the behavior was due to the existence in the meal of some acid of phosphorus beside the orthophosphoric.

Upon testing a large number of samples of meal it was found that the aqueous solutions gave a nearly white precipitate with silver nitrate soluble in nitric acid, and when slightly acidified with acetic acid promptly coagulated a solution of albumen. Magnesia mixture gave a precipitate largely soluble in excess of ammonium chlorid and wholly soluble in excess of magnesium chlorid. About 15 grams of meal No. 408, were dialyzed for twelve hours, the process not being continued longer in order to prevent as much as possible any effects from the decomposition of the organic matter. The diffusate was precipitated with lead acetate, the precipitate thoroughly washed, diffused in a little water and decomposed by sulphuretted hydrogen. The solution was filtered off, freed from sulphuretted hydrogen by a current of carbon dioxid which in its turn was displaced by a current of air. The solution thus obtained was clear and colorless and had a strong acid reaction. It promptly coagulated albumen and gave with silver nitrate no precipitate or a white precipitate soluble in excess of the acid solution. The solution was then nearly neutralized with sodium hydrate. With this solution silver nitrate gave a nearly pure white precipitate, luteocobaltic chlorid a pale, reddish yellow precipitate soluble in excess of the phosphate solution, magnesium sulphate no precipitate, calcium salts a white precipitate soluble in excess of the phosphate. Ammonium molybdate produced a yellow color but only a very trifling precipitate until after the solution had stood for some hours at 60°, when copious precipitation occurred. When the solution of the phosphate was slightly acidified with acetic acid it

promptly coagulated albumen. An aqueous solution of another meal was treated like the foregoing except the sulphuretted hydrogen was displaced from the solution by a current of purified hydrogen gas. Similar reactions were obtained with a small portion of this solution. The rest of it was treated with magnesia mixture and an excess of magnesium sulphate and also with excess of ammonium chloride. The solution was allowed to stand all night and was then filtered from the precipitate. The filtrate was heated to boiling and the precipitate thrown down was collected. The filtrate from this last precipitate was shown to contain a considerable quantity of phosphoric acid, which however, was not immediately precipitated by molybdic solution. The precipitates of magnesium phosphate obtained in the cold and by boiling were dissolved in acetic acid and precipitated by lead acetate. The acids were recovered from these precipitates as already described. The sodium salt formed from the acid thrown down by magnesia mixture in the cold gave all the reactions for orthophosphoric acid. That formed from the magnesia precipitate which occurred on boiling the solution gave a white precipitate with silver nitrate, did not coagulate albumen when acidified with acetic acid, and did not precipitate with ammonium molybdate until after heating and standing. Luteocobaltic chlorid gave a precipitate of the peculiar color of that produced with this reagent by pyrophosphates of the alkalies, although it did not glisten or make its appearance in spangles. I have found, however, that this peculiarity of the precipitate is destroyed by the presence of mere traces of metaphosphate. These reactions all indicate the presence of pyrophosphoric acid. As has already been stated the filtrate from the precipitate in which pyrophosphoric acid was found contained a considerable quantity of phosphoric acid not precipitable by ammonium molybdate until after heating and standing. This filtrate when acidified with acetic acid coagulated albumen, but on account of the salts present neither this test nor the silver nitrate test was of any value. The facts that the original solution of the acids of the meal coagulated albumen, and that phosphoric acid in considerable quantity remained in the solution after removal of ortho and pyrophosphoric acid, and that this phosphoric acid was not readily precipitated by molybdic solution, show that the aqueous solution of cotton-seed meal contains also metaphosphoric acid. In fact all the reactions which have been described seem to show, beyond any reasonable doubt, the presence of both metaphosphoric and pyrophosphoric acid in the aqueous solutions of the meals examined. While no quantitative determinations were attempted, the qualitative results indicated more metaphosphoric than either pyro or orthophosphoric acid.

Two other samples of meal were tested in like manner and with similar results. In the old and very dark meals there appeared to be more orthophosphoric acid and possibly more pyrophosphoric acid than was found in the fresh meals.

Whether pyro and metaphosphoric acid exist in cotton seed or are formed during the preparation of the meal is a point worth investigating. In conclusion it is believed that the failure to obtain all the soluble phosphoric acid in a cotton-seed meal by direct treatment with molybdic solution is due not so much to the presence of the organic matter as it is to the fact that but a small proportion of the phosphoric acid occurs in the tribasic state.

Mr. WHEELER. I have a paper on the use of ammonium nitrate in the determination of phosphoric acid which I beg to present:

THE USE OF AMMONIUM NITRATE IN THE DETERMINATION OF PHOSPHORIC ACID.

By H. J. WHEELER and B. L. HARTWELL.

Svanberg and Struve,[1] Sonnenschein,[2] and others, who first made use of the reaction of ammonium molybdate with phosphoric acid, do not appear to have especially studied the effect of the presence of the various mineral acids and their salts upon the precipitation. R. Fresenius,[3] however, tested the effect of the mineral acids, iron chlorid, and ammonium chlorid. A large excess of nitric acid did not vitiate the results. The results with hydrochloric acid in any considerable quantity were bad. Hydrochloric acid in the presence of nitric acid was even more objectionable than alone. Sulphuric acid, iron chlorid, and ammonium chlorid even in considerable quantities affected the precipitation but slightly. In presence of very large quantities of ammonium chlorid the results were a trifle too low. J. König[4] was led incidentally to pursue the subject further. He found that an addition of 10 grams of ammonium oxalate prevented the precipitation altogether, while 5 and 10 grams additions of ammonium chlorid, ammonium acetate, ammonium sulphate, and ammonium nitrate only slightly affected the results in any case. E. Richter[5] pointed out the difficulty in detecting minute quantities of phosphoric acid in the presence of much free sulphuric or nitric acid or large amounts of their salts, and showed that the presence of potassium, sodium, and magnesium sulphates is more objectionable than their nitrates. Salts of potassium were most objectionable and of magnesium least so. A weak solution of ammonium nitrate (1:20) instead of hindering the precipitation seemed to favor it. The details are given of several experiments with from 0.00002 to 0.00004 grams of phosphoric acid, and in every case he obtained a precipitate by use of ammonium nitrate where without it the solution remained perfectly clear. In experiments with nitrates and sulphates of the alkalis and alkaline earths of a concentration of 1:10, containing 0.00004 grams of phosphoric acid, he used 3 cc. of molybdic solution and 5 cc. of a solution of ammonium nitrate, of which 1 cc. contained 0.15 grams. After some hours precipitation took place even at ordinary temperature, though in the case of the sulphates an extra addition of molybdic solution was found necessary. Without the ammonium nitrate no precipitation took place even after several hours' heating. We have not yet been able to ascertain whether in Richter's original investigation he used a molybdic solution similar to that in use by this association or that used earlier, consisting of ammonium molybdate and pure concentrated nitric acid.

The latter solution contains no ammonium nitrate, while the former solution (100 grams molybdic acid, 400 grams of ammonia of 0.96 sp. g, and 1,500 grams of nitric acid of 1.20 sp. g.) recommended by this association contains, after allowing for the ammonia which would combine with the molybdic acid, about 4.3 grams of ammonium nitrate in every 100 cc.

The methods of analysis adopted by this association for the determination of phosphoric acid prescribe that in case of ignition hydrochloric or sulphuric acid shall be used as a solvent and 15 grams of ammonium nitrate be added in each case before precipitation with molybdic solution. Considering the amount of ammonium nitrate already in the molybdic solution which we employ, the idea suggested itself that possibly the addition of the 15 grams of ammonium nitrate is unnecessary.

---

[1] Jour. f. prakt. Chemie, 44, 291.
[2] Jour. f. prakt. Chemie, 53, 342.
[3] Zeit. f. analyt. Chemie. 3, 446.
[4] Zeit. f. analyt. Chemie, 10, 305.
[5] Dingler's polyt. Jour., Bd. 199 p. 183, abstract Zeit f. analyt. Chemie, 10, 469.

The following results have been obtained by us in the analysis of commercial fertilizers:

Table showing weights of $Mg_2P_2O_7$ obtained.

	Without NH_4NO_3.	With about 7 grams NH_4NO_3.
Phosphoric acid:		
Soluble	.0421	.0420
Soluble	.0283	.0282
Soluble	.0309	.0314
Insoluble	.0125	.0128
Insoluble	.0146	.0144
Total	.0425	.0420
Total	.0378	.0376
Total	.0457	.0457
Total	.0438	.0436
Insoluble	.0208	.0211
Total	.0381	.0384
Insoluble	.0174	.0174
		With about 15 grams NH_4NO_3.
Soluble	.0468	.0470
Soluble	.0479	.0480
Insoluble	.0219	.0221
Total	.0335	.0339
Total	.0593	.0594
Total	.0283	.0279
Soluble	.0466	.0465
Total	.0320	.0317
Total	.0294	.0298
Total	.0358	.0353
Soluble	.0423	.0424

The solutions for total and insoluble phosphoric acid were prepared by incineration and solution in hydrochloric acid. The amount of acid employed varied with the nature of the ash to be dissolved, and the digestion was usually continued for some hours, or until apparently nothing but silica remained undissolved. From 5 to 15 cc. of hydrochloric acid of 1.20 sp. g. were employed for the solution, and the whole made up to 200 cc , 25 cc. being employed in case of total phosphoric acid and 50 cc. for soluble and insoluble. After the addition of the molybdic solution the solutions were digested for from one and one-half to two hours at from 50 to 65°.

These results would indicate that in fertilizer work by the association method, as employed in this work, the addition of ammonium nitrate may, in part or whole, be omitted, and that thus a considerable saving of extra trouble and chemicals may be avoided.

The discussion on the subject of phosphoric acid was directed, first, toward the drying of the samples.

Mr. LORD. I recommend that the sample of 5 grams be dried for five hours in a double-walled water bath three-fourths full.

The PRESIDENT. I ask if other fertilizers, such as potash salts, nitrate of soda, and sulphate of ammonia, should be included in this suggestion.

Mr. LORD. No; only superphosphates. Of these 5 grams should be used; of all other fertilizers, 2 grams.

Mr. SCOVELL. In regard to the temperature, I prefer that of boiling water.

Mr. LORD. The lack of uniformity in temperature comes from ambiguity in the statements of methods which would allow the use of an air bath at different temperatures.

Mr. MCDONNELL. In a jacketed water bath I suggest that the door should be in the top.

Mr. HUSTON. The air bath, instead of the water bath, is used with some materials, and gives good satisfaction. In a double-walled water bath variations in temperature would occur from the size and position of the door. The door of the water bath should be faced with wood, so as to make it nonconducting, and it should fit tightly like a cork.

Mr. PAYNE. A large water bath should be used; one of about 8 gallons is preferred. With this size of bath no trouble was found in securing uniformity of temperature. With a small water bath, especially if water has to be added from time to time, the result is very different.

The PRESIDENT. Please let us know the exact form of the amendment which is proposed.

Mr. LORD. It now reads, "with other fertilizers, heat 2 grams, etc." I propose to change it, and make it 5 grams for five hours in a water oven in which the water is kept boiling continuously. By simply having an extra tube, you can get whatever back pressure is needed. I am able to maintain an average temperature all the time.

The PRESIDENT (to Mr. LORD). Give us the exact form of your proposition.

Mr. LORD. It now reads: "In all other fertilizers heat 2 grams, etc." I propose to have it 5 grams for five hours in a water oven in which the water is kept boiling continuously.

Seconded and adopted.

Mr. DE ROODE. It would be better to leave out the first part of that and put it under the head of potash determinations.

Mr. HUSTON. It seems that this has been compiled from the directions for water determinations in all kinds of fertilizing materials. For the potash salts the instructions should come under the determinations of potash.

Seconded.

Mr. FREAR. I do not believe that most of us have supposed that clause referred at all to complete fertilizers, but rather to separate samples. Every objection can be met by simply saying "moisture determination," and taking that whole section out from under the head of phosphoric acid.

Mr. SCOVELL. If the gentleman will withdraw his motion I will move that we proceed as we did this morning; that we proceed to debate the method on phosphoric acid by the reporter section by section, and adopting or rejecting it, and go on with it.

The PRESIDENT. If that is the wish of the association, we will proceed with phosphoric acid.

Mr. DE ROODE. Do you leave out section 1? I would like to ask what it means by saying (p. 179) "properly prepared." I think we had better have some better direction. It gives no specific direction.

Mr. SCOVELL. I thought that covered the subject. I would leave out that, and then we can put in those two, with general directions.

Mr. PAYNE. I would like to offer a substitute, and have it read, "moisture and sampling," and then go on to phosphoric acid.

Mr. HUSTON. We have already decided about moisture.

Mr. WHEELER. That question has not been decided upon. When the vote was taken it was in relation to the last line, not the question of moisture. I do not think we have had any votes on that proposition.

Mr. HUSTON. I suppose it included it under the head of "heating for five hours."

The PRESIDENT. What is the proposition?

Mr. PAYNE. I offered a substitute for the whole matter; that we have the first head, "moisture and sampling," then phosphoric acid under a separate head.

Mr. DE ROODE. I do not see why moisture should come in with sampling any more than phosphoric acid.

Mr. PAYNE. It covers another ground, I think.

Mr. DE ROODE. Have I a second for my substitute?

The PRESIDENT. It is for the association to say. I do not see myself that we would improve it.

On a vote on the substitute it was lost.

Mr. WILEY. Perhaps it will be well to call attention to the fact that only certain members are entitled to vote. I suggest that those entitled to vote stand up so that the president and secretary may know them. And I think it would be well for the gentlemen discussing these matters to keep a seat in front and as near together as possible.

The PRESIDENT. What Mr. Lord proposed was voted down. Mr. Lord will please repeat it.

Mr. LORD. I proposed it in this way, that for determinations of moisture be substituted "loss on drying," and that it otherwise read: "In all other fertilizers at least 2 grams for five hours in a water oven in which the water is kept boiling continuously."

Mr. KEDZIE. I make the point of order that that proposition has not been placed before the association and adopted.

The PRESIDENT. I beg pardon. I did so understand it.

Mr. FREAR. We can save time and indicate clearly what we mean by using the term "moisture," and I think it is a public advantage, and for that reason I prefer to retain the old term.

Mr. Lord's proposition was then resubmitted to a vote and decided adopted.

Mr. SCOVELL. I move that the report on the determinations of phosphoric acid be taken up, and that as the reporter reads each section by title it be adopted for another year, if there is no objection.

The sections 1 to 4 were read.

Mr. FREAR. In section 4 the directions say, "Raising the temperature as rapidly as practicable to 65°, which is subsequently maintained, digest for thirty minutes from the instant of insertion, shaking every five minutes, filter the warm solution quickly, best with filter pump, and wash with water with about the temperature of 65°."

One would suppose if the water be heated to 65° that the liquid in the flask would in a short time gain the same temperature. I was very much surprised some time ago to find that I could not get, on any reasonable length of heating, 65° inside the flask when I had 65° outside. I did not know exactly the cause of it. The flask was so placed that the water stood a little above the level of the liquid inside the flask.

Mr. WHEELER. My idea would be to raise the citrate to 65° and then insert the substance and maintain it at 65° for half an hour, and the results would be more uniform than they can be in any other way.

The PRESIDENT. In my laboratory the way is to raise the temperature to 65° and keep it as uniform as possible.

It seems to me that the language of the directions is sufficient: "Cork the flask securely and place it in a water bath, the temperature of which stands at 65°."

Mr. WHEELER. I believe that there will be a very little loss of moisture if the citrate solution were brought to a temperature as rapidly as possible, and then insert the substance and cork it up and hold it there for half an hour.

Mr. FREAR. I wish to propose this amendment, to insert the words "of the bath" so that it will be read, "raising the temperature of the bath as rapidly as practicable to 65°."

Seconded.

Mr. PARSONS. I would like to say in reference to the point of the flask not being up to 65°, that it makes a difference what kind of a bath is used, and whether it is on a level with the water in the flask or not. Recently we have put in a new bath by which the flask is submerged so that the water surrounds it up to the neck. There is a large amount of water, so that putting in the flask does not change the temperature more than from 2° to 3°. Under those conditions we have kept the temperature inside of the flask very nearly to 65°.

Mr. LORD. I think it will be well to remember that the method as used has been pretty largely tried, and this proposition for a change, I think, should be placed on experimental work. I think we should avoid modifications unless they are established by experience.

The PRESIDENT. The question before us is on Mr. Frear's motion to add the words, "of the bath," as stated by him.

Carried.

Mr. LORD. I propose to omit "in case of goods containing iron or alumina, the addition of a small amount of citrate solution to the wash water will give clear washings."

Mr. HUSTON. I will be responsible for that statement, but am not at all desirous that it should stand.

Mr. LORD. My motion is to strike that out.

Mr. HUSTON. I am willing that it go out.

It was so ordered.

With other minor changes, which appear in the methods of analysis, the method of phosphoric acid analysis was adopted as a whole for official use from time of publication.

On motion the association adjourned at 5 p. m., August 25, to meet at 9:30 a. m., Friday, August 26, 1892.

SECOND DAY.

MORNING SESSION, FRIDAY.

The convention was called to order by the president at 9:30 o'clock.

Mr. Payne presented the following report on potash:

REPORT ON POTASH.

By Dr. GEORGE F. PAYNE, *Atlanta, Ga.*

The work required by the association, of the reporter on potash for 1892, was an investigation as to whether the use of sodium chlorid was essential in the Lindo-Gladding method of determining potash.

Four samples were prepared and were numbered 1, 2, 3, and 4.

No. 1 was a high grade sulphate of potash.

No. 2 was an acid phosphate with sulphate of potash in the form of kainit.

No. 3 was an acid phosphate with sulphate of potash in the form of kainit, and containing 5 per cent of double sulphate of alumina and ammonia.

No. 4 was identical with No. 3 with the substitution of 5 per cent of soluble sodium silicate in place of the double salt of alumina and ammonia.

The ingredients of each sample were first finely powdered and thoroughly mixed, then sifted through a very fine sieve, then again intimately mixed and portions taken from forty different parts of the pile were well remixed and used for filling the bottles. Only the best XX superfine long taper corks were used and were selected long enough to project well from the mouth of the vial to secure easy opening and air-tight recorking. The corks were all well sealed with paraffin. With each package a circular was sent requesting that the determinations be made as soon as convenient after receiving samples to eliminate as far as possible any differences likely to occur from changes in the amount of moisture after bottling.

Each chemist was also requested to carry out the method as ordinarily managed in his laboratory, working each sample with and without sodium chlorid, side by side, and as nearly as possible under exactly the same conditions.

Twenty-two chemists took part in the work and their results were as follows:

Determination of potash by the Lindo-Gladding method with and without sodium chlorid.

Names of analysts and location.	Sample No. 1.		Sample No. 2.		Sample No. 3.		Sample No. 4.	
	With NaCl.	Without NaCl.	With NaCl.	Without NaCl.	With NaCl.	Without NaCl.	With NaCl.	Without NaCl.
OFFICIAL LABORATORIES.								
Norman Robinson, State chemist, Tallahassee, Fla	48.83	48.70	1.58	1.59	1.47	1.46	1.40	1.41
F. S. Shiver, assistant of State chemist, Fort Hill, S. C (a)	49.05	49.13	1.45	1.44	1.40	1.39	1.40	1.44
F. S. Shiver, assistant of State chemist, Fort Hill, S. C (b)	49.00	.....	1.45	.....	1.40	.....	1.32	.....
P. L. Hutchinson, assistant of State chemist, Atlanta, Ga	49.05	49.15	1.50	1.48	1.51	1.40	1.48	1.40
J. F. Wilkinson, assistant of State chemist, Atlanta, Ga	49.13	49.12	1.50	1.52	1.47	1.42	1.36	1.34
F. B. Carpenter, Experiment Station, Raleigh, N. C	48.94	.....	1.61	.....	1.48	.....	1.56	.....
Thos. L. Blalock, Experiment Station, Raleigh, N. C (a)	48.76	48.76	1.63	1.63	1.48	1.46	1.48	1.47
Thos. L. Blalock, Experiment Station, Raleigh, N. C (b)	48.96	49.10	.....	.....	.....	.....	.....	.....
Thos. L. Blalock, Experiment Station, Raleigh, N. C (c)	48.90	48.91	1.59	1.58	1.51	1.48	1.46	1.45
Louis A. Voorhees, Experiment Station, New Brunswick, N. J	49.23	49.19	1.49	1.46	1.35	1.36	1.36	1.37
Louis A. Voorhees, Experiment Station, New Brunswick, N. J	49.27	49.29	1.51	1.52	1.38	1.42	1.36	1.37
Chas. S. Cathcart, Agricultural College, New Brunswick, N. J	49.31	49.34	1.52	1.46	1.43	1.45	1.35	1.33
E. H. Farrington, Experiment Station, Champaign, Ill	49.29	49.00	1.77	1.64	1.77	1.66	1.62	1.51
Thos. L. Watson, Experiment Station, Blacksburg, Va., assistant chemist	49.19	48.95	1.55	1.58	1.41	1.42	1.41	1.36
R. C. Kedzie, Agricultural College Post-office, Mich	48.75	48.69	1.55	1.53	1.54	1.51	1.67	1.65
Rudolf de Roode, Experiment Station, Morgantown, W. Va	48.96	48.96	1.52	1.55	1.42	1.42	1.37	1.39
Rudolf de Roode, Experiment Station, Morgantown, W. Va	49.04	49.04	1.57	1.53	1.48	1.46	1.39	1.37
C. B. Collingwood, Experiment Station, Tucson, Ariz	48.62	48.38	1.51	1.47	.....	.....	.....	.....
Henry E. Curtis, Experiment Station, Lexington, Ky	49.00	48.91	1.45	1.46	1.43	1.40	1.39	1.37
W. S. Sweetser, Experiment Station, State College Post-office, Pa	49.26	49.28	1.69	1.57	1.58	1.53	1.45	1.43
W. S. Sweetser, Experiment Station, State College Post-office, Pa	49.30	49.29	1.62	1.57	1.51	1.51	1.45	1.40
H. B. McDonnell, State chemist, College Park Post-office, Md	48.47	48.69	1.65	1.61	1.58	1.55	1.48	1.45
W. D. Bigelow, U. S. Department of Agriculture, Washington, D. C	48.56	48.31	1.49	1.58	1.57	1.37	1.47	1.42
Average	48.994	48.961	1.554	1.538	1.484	1.456	1.439	1.417
Difference	.....	.033	.....	.016	.....	.028	.....	.022
UNOFFICIAL LABORATORIES.								
Shepard Laboratory, Charleston, S. C	49.88	49.80	1.87	1.91	1.75	1.74	1.66	1.55
Elwyn Waller, School of Mines, Columbia College, New York City	49.67	49.17	1.60	1.46	1.61	1.53	1.59	1.59
Elwyn Waller, School of Mines, Columbia College, New York City	49.89	48.71	1.62	1.52	1.56	1.52	.....	1.53
Elwyn Waller, School of Mines, Columbia College, New York City	.....	48.21	.....	.....	.....	.....	.....	.....
Elwyn Waller, School of Mines, Columbia College, New York City	.....	48.69	.....	.....	.....	.....	.....	.....
H. T. Vulte, assistant of Elwyn Waller, School of Mines, Columbia College, New York City	48.88	48.54	1.66	1.58	1.58	1.53	1.58	1.52

Determination of potash by the Lindo-Gladding method with and without sodium chlorid—Continued.

Names of analysts and location.	Sample No. 1.		Sample No. 2.		Sample No. 3.		Sample No. 4.	
	With NaCl.	Without NaCl.	With NaCl.	Without NaCl.	With NaCl.	Without NaCl.	With NaCl.	Without NaCl.
UNOFFICIAL LABORATORIES—continued.								
B. C. Hinman, assistant of Elwyn Waller, School of Mines, Columbia College, New York City	.....	48.99	.....	1.51	1.67	1.40	1.41	1.40
F. S. Hyde, student of School of Mines, Columbia College, New York City	49.43	49.47	1.35	1.42	.....	.....	.....	.....
Average	49.550	48.948	1.620	1.566	1.634	1.544	1.560	1.518
Difference	.....	.602	.....	.054	.....	.090	.....	.042
Total average	49.093	48.957	1.566	1.545	1.513	1.474	1.458	1.438
Difference	.....	.136	.....	.021	.....	.039	.....	.020

NOTES FROM CORRESPONDENTS.

NORMAN ROBINSON (Florida).—As to the addition of NaCl, I have long since discarded it in regular laboratory work, as I was never able to satisfy myself that it was of enough benefit to pay for the additional expense in platinic chlorid.

F. S. SHIVER (South Carolina).—Determinations on line "a" were made by a modification of the Lindo-Gladding method, as follows: "After $PtCl_4$ has been added the solutions are evaporated to dryness on the water bath, which is heated not quite to boiling. A little water is now added, about 1 cc. in case of superphosphates, and about 2 cc. in case of muriates, sulphates, and kainits. Five volumes of 95 per cent alcohol for each one of water are now introduced; the whole thoroughly mixed and allowed to stand one hour. Filtration and washing with 80 per cent alcohol and the NH_4Cl solution follows in the usual way." Results on the line "b" were obtained by strictly following the official method.

THOS. L. BLALOCK (North Carolina).—Results on line "a" and "b" were obtained by evaporating until crystallization took place on cooling. Those on line "c" were evaporated to dryness and moistened with water.

W. S. SWEETSER (Pennsylvania).—I find that when NaCl is used more washing is required in order to get uniform results.

H. B. MCDONALD (Maryland).—The use of NaCl is certainly unnecessary.

ELWYN WALLER (New York).—Some of the results may differ from others on the same sample because of differences in percentage of moisture in the sample at different times, but the conclusion I should make is that without NaCl there was a tendency to irregularity, and on the whole, lower results. The direction for the Lindo-Gladding method in the Bulletin does not seem to give the idea as to the amount of ammonium oxalate required. It says, "a little." I find that 10 grams needed over 50 cc. of (1:24) solution for complete separation of CaO. I analyzed a pure substance by the same methods (theory=9.90 per cent); result with NaCl=9.816; result without NaCl=9.836, which seems to be essentially the same.

H. J. WHEELER (Kingston, R. I.).—I am very glad you are to take up the point in question, for past results indicate the possible benefits from such a course.

W. J. WILLIAMS (Wilmington, Del.).—I may state that on several occasions I have tested this question and have found that if the potash after treatment with half-strength sulphuric acid is thoroughly ignited to whiteness, so that no free sulphuric acid is left, the addition of NaCl makes no more difference in results than will be found in ordinary analytical differences. I believe it is unnecessary.

The mass of evidence here presented furnishes strong and convincing proof that the use of NaCl is unnecessary in potash determinations.

Dr. Gladding in 1885 presented his results to his association upon chemically pure potassium sulphate. Three determinations without NaCl gave him 100.95, 102.81, and 101.45 per cents respectively of potassium sulphate. Three determinations of the same salt made with the addition of NaCl gave 100.01, 100.16, and 99.95 of potassium sulphate, obviating the inaccuracy he supposed to exist from the presence of free sulphuric acid when the NaCl was not used.

Two hundred and nine results obtained by twenty-two chemists show no advantage in the use of NaCl. Instead of obtaining something which was not potash and hence higher figures, without NaCl, the general tendency has been to lower results when NaCl was omitted. This difference was quite small, but is present in a large majority of the determinations and is naturally to be expected, if the NaCl is not needed, on account of the difficulty of washing out the sodium salt. About two-thirds of the analysts who took part in the work obtained slightly higher figures with NaCl than without it, the reverse of Dr. Gladding's experience. In about three-fourths of the determinations the differences between with and without NaCl, by the same chemist, were less than one-tenth of 1 per cent, and in about half of the results the differences were less than one-twentieth of 1 per cent.

RECOMMENDATIONS.

The following recommendations are made:

In superphosphates, page 187, second line, in place of the words "a little ammonium oxalate" insert "a sufficient quantity of ammonium oxalate to throw down all lime present."

On lines Nos. 9 and 10 strike out the words "5 cc. of a solution of pure NaCl containing 20 grams NaCl to the liter, or its equivalent of dry salt."

On line No. 11 change the word "are" to the word "is."

Sulphate of potash, kainit, etc., page 187, second and third line, in place of the words "a little ammonium oxalate" insert "a sufficient quantity of ammonium oxalate to throw down all lime present."

On page 188, second line from top, strike out the words "add 0.25 grams of NaCl" and insert the word "add" between "and" and "15 cc."

On page 188, fifth line from top, strike out the words "0.25 grams NaCl and."

Page 188, fifth line. Put the word "all" in place of the word "these" and in the seventh line, after the words "to remove all the double platinum-sodium chlorid," add the words "if present."

It is also recommended that the reporter on potash for the coming year put himself in communication with the Association of Experiment Stations in Germany and any accessible official chemists in other countries and secure, as far as possible, their coöperation in our comparative work of the coming year.

Bearing on this subject the following quotations are translated from a letter of the German Kali syndicate of July 29, 1892:

"As yet, the Association of Experiment Stations of the German Empire has not accepted any definite methods for potash determination. Not many stations use the Stassfurt methods. A variety of methods are in use, according to the preference given by different chemists. We have made a request of the Association of Agricultural Experiment Stations of the German Empire to take steps for securing uniformity in this question. Privy Counselor Maercker, who is president of the committee on fertilizers, has already declared that he will make a motion at the next convention in September to test the Stassfurt methods and eventually to recommend their introduction."

Further on will be found the methods of potash determination used at the Anhalt mines at Stassfurt, Germany, as communicated by their chief chemist, Dr. Zimmermann, with his criticisms of other methods. His work, while most excellent with the Stassfurt method, shows want of experience with the Lindo-Gladding method, as in these analyses the difference between the highest and lowest is over 1 per

cent, a difference for which the method is certainly not to blame, when so many American official chemists are constantly working it with results equally as close as he obtains with the Stassfurt method.

We wish accurate methods and quick ones and are always ready to grasp anything good. The Stassfurt method is submitted to the association with the suggestion that it be taken up and studied through our next report on potash. If it is equally as good as the Lindo-Gladding and much quicker, as claimed, it will be very valuable.

The London Chemical News of January 8 and 15, 1892, contains an interesting article by Drs. Gooch and Hart upon the spectroscopic detection and estimation of potassium.

There is a point in the determination of potash which is not given the proper weight and attention by manufacturers, and it is that of mixing. Unmanipulated potash salts give analytical chemists far less trouble, as a rule, than mixed fertilizers.

To mix 50 pounds of rough muriate of potash with a ton of fertilizer, as is sometimes done, and secure a perfect mixture, is almost impossible. Weights and measures are handled roughly and quickly, and some manufacturers appear to believe that badly mixed goods, after standing and becoming disintegrated by the sulphuric acid, actually become well mixed. If a solution is made up from a fertilizer and several determinations are made from that same solution, they tally beautifully; but make separate weighings from your supposed well-mixed sample and results never check so well. Manufacturers think a "wet mix" ought to be perfect, but as all the ingredients are weighed independently for each ton, and in the South negroes usually do this work, and bricks, in some establishments, are used as weights, it is easy to see how each ton must vary, and the only wonder is that the goods run as uniformly as they do. To illustrate this fourteen analyses are here given of goods made by the same formula and claimed to be identical, which were drawn out of separate batches from a large and well-equipped factory. These samples show imperfect mixing, whether from too rough weighings or bad manipulation it is difficult to decide, with only small samples drawn by other parties. Attention is also called to the varying amounts of moisture, insoluble phosphoric acid, and ammonia. Moisture varies 8.30 per cent, insoluble phosphoric acid 4.13 per cent, ammonia .77 per cent, and potash .95 per cent.

Moisture.	Insol. P_2O_5.	Avail. P_2O_5.	Ammonia.	Potash.
14.95	2.33	9.95	2.37	2.16
15.00	2.24	10.30	2.29	2.19
13.50	4.22	9.34	2.26	2.03
16.85	1.60	10.17	2.33	2.24
16.55	2.49	9.41	2.29	2.12
16.60	2.43	9.21	2.25	2.20
16.55	2.62	9.41	2.12	1.70
16.25	2.36	9.79	2.00	1.80
17.75	1.00	10.90	1.93	1.60
18.85	.41	9.82	2.33	2.33
19.45	.26	10.11	2.53	2.30
21.80	.09	9.76	2.44	2.55
19.05	.90	9.46	2.50	2.35
18.10	.64	10.11	2.70	2.45

Methods of potash analysis as used in the mines of Anhalt.

(1) Determination of potash alone in carnallit, kainit, and sylvinit: 100 grams of the well-mixed sample are put into a graduated flask holding 1,000 cc. and dissolved by boiling with 500–600 cc. water, acidulated with 10 cc. hydrochloric acid. The purpose of adding hydrochloric acid is to bring polyhalit in solution, that might be present in the salts and which it is difficult to dissolve in pure water. After dissolving and cooling, the flask is filled up to the mark. The solution after thorough mixing is filtered through a dry filter and 100 cc. of the filtrate, corresponding to 10 grams substance, are put into a 500 cc. flask by means of a pipette. After the addition of 200–300 cc. water, the solution is heated to boiling and the sulphuric acid accurately precipitated with normal barium chlorid solution, containing 104 grams of the dry salt in one liter. The volume of the precipitate is calculated from the amount of barium solution used and from the specific gravity of the barium sulphate. After cooling, the flask is filled up with water so much above its mark as equals the volume of the calculated barium precipitate, and, after thorough mixing, the solution is filtered again through a dry filter. Fifty cc. of this filtrate, corresponding to 1 gram substance, are evaporated upon the water bath with a sufficient amount of platinum chlorid. The residue of potassium-platinum chlorid is washed with 90 per cent alcohol, dried at 120°, and weighed.

(2) If it is desired to determine separately the quantity of potash present in the form of sulphate and in the form of chlorid, as for example in kainit and in sulphate of potash, or if it is to be determined whether potassium sulphate is in combination with a proportionate amount of magnesium chlorid, as in kainit, or in combination with magnesium sulphate alone, as in schönit, it becomes necessary to determine besides potash the percentage of chlorin, sulphuric acid, lime, magnesia, the total alkalis, water, and the residue, insoluble in water. For this purpose 100 grams of the sample are dissolved, the solution is filtered, the filter washed, and the filtrate made up to one liter; a part of the liquid is taken for the determination of sulphuric acid, by precipitating with barium chlorid, and another part for the determination of lime and magnesia. For the determination of the alkali chlorids, 100 cc. of the solution, corresponding to 10 grams substance, are acidulated with hydrochloric acid, and, after heating to boiling, the sulphuric acid is completely precipitated with barium chlorid, with the precaution of using not more of the barium solution than is necessary for the complete precipitation. Fifty cc. of the filtered solution, corresponding to 1 gram substance, are evaporated to dryness, in order to drive off the hydrochloric acid. Magnesium chlorid is decomposed by igniting with oxalic acid or with mercuric oxid. After ignition, the residue is moistened with a little ammonium carbonate for the purpose of rendering the calcium oxid, that may have been formed, into calcium carbonate. The alkali chlorids, which are now entirely free from lime and magnesia, are weighed, and potassium chlorid is determined by means of platinum chlorid. The amount of sodium chlorid results by deducting potassium chlorid from the mixed chlorids. For the water determination 5 grams of the sample are ignited and the loss of weight is determined. The ignited mass is dissolved in water, and for the purpose of determining the quantity of magnesium chlorid that may have been decomposed by the ignition the percentage of chlorin is determined by titration. The difference in the contents of chlorin before and after ignition is subtracted from the loss in weight, after allowance has been made for the absorption of oxygen and for the loss of hydrogen. The balance is water. The results obtained are calculated in the following manner: From the total amount of the sulphuric acid found, that portion is deducted which is combined with calcium as calcium sulphate, the balance of the sulphuric acid is divided into two equal parts, for the purpose of calculating the contents of potassium sulphate and magnesium sulphate, according to the molecular proportion in which these salts are present in kainit and in schönit. If there

is an excess of potash left uncombined with sulphuric acid, then it is in the form of potassium chlorid; likewise the amount of magnesia, uncombined with sulphuric acid, is to be reckoned as magnesium chlorid. The result of this calculation will tell how much potash is in the form of kainit (K_2SO_4, $MgSO_4$, $MgCl_2$ with $6H_2O$) and how much of it is in the form of schönit (K_2SO_4, $MgSO_4$, with $6H_2O$) and in the form of potassium chlorid. The sodium is reckoned as sodium chlorid.

(3) In calculating the contents of potash, of potassium chlorid and of potassium sulphate from the weighed potassium-platinum chlorid, the factors 0.1928, 0.3056, and 0.3566 are used, assuming that the atomic weight of platinum is 197.18.

(4) The two methods which have been described under Nos. 1 and 2, and which are in common use in the Stassfurt potash industry, *i. e.*, the so-called precipitation method and the oxalic-acid method, give almost identical results. The first method, however, deserves preference on account of greater simplicity in cases where potash alone is to be determined. Finkner's method likewise gives results which agree well with the results obtained by the customary methods. It consists in evaporating the salt solution with a sufficient quantity of platinum chlorid without previously removing the sulphuric acid, reducing the potassium-platinum chlorid, and weighing the metallic platinum.

The following are the results of comparative analysis, executed by order of the manufacturers of muriate of potash. The sample investigated was taken from a quantity of 86,948 cwts. of carnallit; the following are the results:

1. After the precipitation method..... 22.02 per cent KCl
2. After the oxalic-acid method..... 22.03 per cent KCl
3. After Finkner's method..... 22.01 per cent KCl

In another sample from 75,582 cwts. carnallit the following results were obtained:

1. After the precipitation method..... 17.88 per cent KCl
2. After the oxalic-acid method..... 17.88 per cent KCl

In a third sample from 97,435 cwts. of carnallit the content of potassium chlorid was as follows:

1. After the precipitation method..... 18.44 per cent
2. After the oxalic-acid method..... 18.38 per cent

Another method of potash determination which is used in many agricultural experiment stations, among them the royal station of Hohenheim in Würtemberg, consists in precipitating the sulphuric acid and alkaline earths with barium oxid and ammonium carbonate, and potash with platinum chlorid. The results obtained with this method are, according to our experience, very inaccurate, and always too low. This is explained by the fact that it is impossible to precipitate sulphuric acid without at the same time precipitating some of the potash, unless it be in an acid solution.

A separation of the alkaline earths, if potash alone is to be determined, is superfluous, for the reason that calcium and magnesium-platinum chlorid are soluble in 90 per cent alcohol, even with more facility than sodium-platinum chlorid.

(7) There is a method in use and recommended by the Association of Official Agricultural Chemists of the United States of North America, termed the Lindo-Gladding method, for analysis of potash salts, which has been reported to us by Dr. B. von Herff, of Washington. We subjected this method to a thorough test in our laboratory; the results obtained do not agree with those obtained by our customary methods, nor do they agree among themselves, if repeated analyses of the same sample are made. Moreover this method is considerably more troublesome and subject to more errors than the precipitation method. An average sample of a large cargo contained:

1. AFTER THE STASSFURT METHOD.	2. AFTER THE LINDO-GLADDING METHOD.
1. 12.92 per cent K_2O.	1. 13.47 per cent K_2O.
2. 12.97 per cent K_2O.	2. 12.39 per cent K_2O.
3. 12.94 per cent K_2O.	3. 13.29 per cent K_2O.

Likewise the results of repeated analyses of a sample from a large quantity of sulphate of potash gave according to the precipitation method: 52.7 per cent potash, while three determinations after the Lindo-Gladding method gave: 53.40 per cent, 53.03 per cent, and 53.61 per cent. These figures presented will amply demonstrate the unreliability of this method.

Mr. WILEY. I am sorry that I do not see Mr. von Herff here, as I am sure he had something to present on the subject of potash. He is a representative of the German Kali Company at the present time. He spoke to me the other day about the propriety of his making the presentation of the German methods of potash analysis, and I told him I thought we had no objection to it whatever. Mr. Payne, however, has discussed these methods in his report.

Mr. PAYNE. There is one thing I want to call attention to. I have a paper that was handed me yesterday afternoon from the New York State Agricultural Experiment Station, which I overlooked. It gave 191 results on determinations of potash, and the results correspond with the results we have obtained by the official chemists' method practically. There were 88 cases that were higher without the use of salt and 95 that were lower, showing that they washed out the salt pretty well. Eight gave the same results by both methods. There seems to be a disposition in Germany to compare their work with ours, and I think it would be a very good plan to do so. They have a number of experiment stations in Germany and we would like their coöperation in comparative work the coming year.

The PRESIDENT. If there are no other papers to be presented on this subject, I will submit the matter for your action.

Mr. PAYNE. There is one recommendation, that is, the last recommendation on page 188 of last year's bulletin, in the sixth line, "to remove all the double chlorid of platinum and sodium," that I think ought to be left in. I should add that I got this thing up in a hurry.

Mr. KEDZIE. I move that the first recommendation be adopted as read.

Adopted.

The additional recommendations of the reporter were adopted as read.

Mr. MCDONNELL. I move that the words "to remove all the double chlorid of platinum and sodium," on page 188, be stricken out. I think they are unnecessary.

Mr. WILEY. We might add the words "if present."

Mr. MCDONNELL. I am willing to accept that.

The president submitted the motion of Mr. McDonnell as amended by Mr. Wiley, and declared it carried.

Mr. DE ROODE. The quantity of platinum solution used in both cases generally is excessive and I think it would be well to change that somewhat. I recommend that this be changed to make the solution of such strength that 1 cc. precipitates about 1 per cent of potash. Thus you save the trouble of working over the platinum too often.

The PRESIDENT. The platinum solution has become quite an item now.

Mr. DE ROODE. Yes. We do not lose much of it, but save the trouble of working the platinum over too often.

Mr. HUSTON. In the description of making up the platinum solution, I move that instead of saying 10 grams of metallic platinum say 4 grams of metallic platinum, "and make a note after that saying" 1 cc. of this solution may be taken for each per cent of potash.

Mr. DE ROODE. I withdraw my amendment and move to amend as follows: "Instead of saying 4 cc. platinum solution" say "sufficient to precipitate all potash present."

Mr. PARSONS. We have also got to precipitate the sodium and ammonium present. That fact ought to be brought out.

Mr. WHEELER. This seems to me wholly unnecessary, because in precipitating a potash solution we will calculate the strength of the platinum and add enough to cover the case in question. If one is working with 2 per cent solution or 10 per cent he can use his judgment in that. It seems to me that is going outside of our real office. The man must use his judgment.

Mr. DE ROODE. I move to amend as follows: Before the word "excess," insert the word "slight" and strike out the 4 cc. in the parenthesis altogether.

The president submitted the motion of Mr. De Roode and declared it adopted.

Mr. WILEY. Before leaving this section and passing on to the "Alternative methods," I call attention to the fact that on page 188, under the head of "Alternative methods," you are directed to prepare the fertilizer as for the Lindo-Gladding method. If you turn back to the Lindo-Gladding method you will see nothing at all about preparing the fertilizer; therefore the person who refers to this method for directions to prepare this sample will be somewhat in the dark.

The PRESIDENT. Yes. I was looking that up myself, and I could not see that we had given directions.

Mr. WILEY. I only wanted to hear what the reporter had to say in regard to preparing the samples.

Mr. PAYNE. None of the association noticed it last year when it was adopted.

Mr. SCOVELL. Yesterday we arranged for the preparation of the samples under the head of "Fertilizers."

Mr. WILEY. Better change the reference.

Mr. SCOVELL. On page 179 you will find a section headed "Preparation of samples."

The PRESIDENT. It is moved and seconded as follows: "Take 10 grams, boil for 10 minutes with 300 cc. of water, and after cooling and without filtering the solution make up to 1,000 cc. and filter through a dry filter," etc.

Adopted.

Mr. KILGORE. On page 187, fourth line of potash methods, I move to insert the word "cool" before "make up to 500 cc."

Adopted.

Mr. WOODS. Would it not be well to make the same change in the second or third line from the bottom of the page?

The PRESIDENT. If there is no objection it will be so considered.

Mr. HUSTON. I would like to call attention to the fact that in fertilizers containing a large amount of tobacco stems, the phosphoric acid present is liable to be reduced in the ignition with sulphuric acid and to attack the platinum. I have had a good deal of trouble when working with such mixtures. I understand that a good many don't use platinum. We have always used platinum. We have made a number of trials and I intended to bring my figures along; but, unfortunately, I left the notebook at home.

We have added a little calcium chlorid to unite with the phosphoric acid, and then we can use platinum without danger. In case one uses platinum I thought I would simply say that we have made repeated determinations using calcium chlorid and without, and the results are practically the same by both methods. I find it is very convenient provided enough calcium chlorid is added to precipitate all the soluble phosphates present.

The methods for potash were then adopted as given on page 197.

Mr. Van Slyke was called upon and read his report, which was as follows:

REPORT ON METHODS FOR THE DETERMINATION OF NITROGEN.

By L. L. VAN SLYKE.

The special instructions which the association gave, by vote at its last meeting, to the reporter on nitrogen were as follows:

"To investigate the methods of standardizing acids in general and to pay special attention to the tetra-oxalate method (pp. 154, 155, Bull. 31); to test the various indicators with reference to this subject; to look into the advisability of adopting the method of De Roode of determining nitrogen and phosphoric acid in the same weighed portion of substance (p. 111, Bull. 31); to make a thorough test of the Gunning method (p. 143, Bull. 31)."

As the work relating to the De Roode method belonged more properly to the reporter on phosphoric acid I arranged with him to include it in his investigation. The reporter on nitrogen for 1891 made several suggestions in his report for special lines of future work. I decided to adopt one of these suggestions relating to the determination of nitrogen in the form of pure nitrates by the Ulsch method. In continuation of some of last year's work and at the special suggestion of others I decided to add a comparison of the use and omission of potassium permanganate in the Kjeldahl and Gunning methods.

A limited number of invitations was sent out asking for coöperation in the whole work, while a large number of analysts were asked to participate only in the comparison of methods.

For comparing methods three samples were distributed. They were as follows: No. 1, consisting of bone meal; No. 2, consisting of cotton-seed meal; and No. 3, consisting of pure sodium nitrate. Samples No. 1 and No. 2 were used in comparing

the Kjeldahl and Gunning methods and in studying the effect of the use and omission of permanganate in these methods. No. 3 was used for testing the Ulsch method for determining nitrogen in the form of pure nitrates. A sealed bottle of standardized hydrochloric acid was also sent for the purpose of comparing it with the standard acid of each analyst. For the work on tetra-oxalate a sample obtained from Baker & Adamson was sent out. Samples Nos. 1 and 2 were prepared by passing through sieves containing 50 meshes to the inch. The sample of sodium nitrate was finely powdered and dried for 36 hours over sulphuric acid before bottling.

Moisture.

In order to insure uniformity of samples, in case of any possible change before analysis, each analyst was asked to dry 2 grams of samples Nos. 1 and 2 in air for four hours at 100°. This was done by most of the analysts and the results were sufficiently concordant to show that the samples had not changed since leaving the reporter's hands. The analytical results reported are, therefore, based upon the original substances.

Comparison of the Kjeldahl and Gunning Methods.

The following instructions were given to each analyst:

(1) Make duplicate determinations of nitrogen in samples Nos. 1 and 2, using about 1 gram of material for each determination by the Kjeldahl method as described on page 184 of Bulletin No. 31.

(2) Make duplicate blank determinations by the same method.

(3) Make duplicate determinations of nitrogen in samples Nos. 1 and 2 by Gunning's method; as follows:

In a digestion flask holding from 250 to 500 cc., place about 1 gram of the substance, then add 10 grams of powdered potassium sulphate and from 15 to 25 cc. of concentrated sulphuric acid. Conduct the digestion as in the Kjeldahl process, starting with a temperature below boiling point and increasing the heat slowly until frothing ceases. Digest until the solution is colorless or nearly so; do not add either potassium permanganate or potassium sulphid; dilute, neutralize, and distill as in the Kjeldahl method. In neutralizing, it is convenient to add a few drops of phenolphthalein indicator, by which one can tell when the acid is completely neutralized, remembering that the pink color, which indicates an alkaline reaction, is destroyed by a considerable excess of strong fixed alkali. In distilling, the use of zinc or of any substance to prevent bumping or foaming is generally unnecessary, if too great an excess of fixed alkali is avoided. The amount of sulphuric acid recommended by Gunning is 2 grams for each gram of potassium sulphate; but in my experience I have found that this mixture is so viscous as to cause troublesome foaming frequently, and after cooling cakes in a hard mass, which may give trouble to redissolve. To avoid foaming and caking, I have found it an effective means to increase the amount of sulphuric acid used, taking instead of 2 grams to 1 of potassium sulphate 3 or 4 grams of acid to 1 of potassium sulphate. It is, therefore, suggested that, in carrying out the work, you use from 15 to 25 cc. (ordinarily about 20 cc.) of sulphuric acid for 10 grams of potassium sulphate. In case the potassium sulphate is not free from nitrogen compounds, one or two recrystallizations will make it pure.

(4) Make duplicate determinations of blanks, using only sulphuric acid and potassium sulphate in digestion.

(For original paper on the Gunning method, see Zeitschrift für analytische Chemie, Vol. 28, pp. 188 to 191.)

Before presenting the results of the work, it may not be out of place to touch briefly upon the chemical reactions involved in the Gunning modification of the Kjeldahl method.

The first reaction to take place is the union of sulphuric acid and potassium sulphate to form potassium acid sulphate in accordance with the following equation:

$$(1)\ K_2SO_4+H_2SO_4=2KHSO_4.$$

When heated, the potassium acid sulphate decomposes, forming potassium disulphate and water, thus:

$$(2)\ 2KHSO_4=K_2S_2O_7+H_2O.$$

The potassium disulphate at a higher temperature decomposes, forming normal potassium sulphate and sulphur trioxid, thus:

$$(3)\ K_2S_2O_7=K_2SO_4+SO_3.$$

At a sufficiently high temperature the two preceding reactions may take place in one, thus:

$$2KHSO_4=K_2SO_4+H_2O+SO_3.$$

At the temperature at which these reactions take place, the water that is set free does not recombine with the sulphur trioxid nor with the sulphuric acid that is present in excess, but is expelled from the mixture; hence the mixture becomes more concentrated during the digestion. The sulphur trioxid set free acts upon the organic matter in the powerful manner peculiar to it, and the potassium sulphate formed in the last reaction above unites with another molecule of sulphuric acid, and the same round of reactions is repeated continuously so long as there is an excess of free sulphuric acid present in the mixture. As the liquid becomes more concentrated with the continuation of the digestion, the boiling point increases so that the effect is the same as heating under pressure. The danger of too great concentration and risk of consequent loss of nitrogen is avoided by using the increased proportion of sulphuric acid recommended above.

As compared with the Kjeldahl method, the Gunning method presents the following advantages:

(1) The Gunning method requires fewer reagents. As no form of mercury is used no potassium sulphid is needed, and there is no risk of loss from the presence of mercurammonium compounds.

(2) The solution to which caustic soda is added is clear, so that in neutralizing it is an easy matter to avoid great excess of alkali, and so, in most cases, to avoid foaming and bumping in distillation.

(3) In the blanks reported by the analysts, less nitrogen was found in the reagents used in the Gunning method. In only one case was more nitrogen reported in a blank by this method: in all the others the amount averaged considerably less.

Comparison of the Kjeldahl and Gunning methods.

State.	Analyst.	Bone meal.			Cotton-seed meal.		
		Kjeldahl method.	Gunning method.	Difference.	Kjeldahl method.	Gunning method.	Difference.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per ct.*
Arkansas	G. L. Teller	4.41	4.39	0.02	7.15	7.15	0.00
Illinois	E. H. Farrington	4.46	4.42	0.04	7.17	7.09	0.08
Kentucky	H. E. Curtis	4.50	4.46	0.04	7.27	7.22	0.05
Michigan (University)	E. R. Miller	4.44	4.44	0.00	.....	.....	.....
New Jersey	E. B. Voorhees	4.52	4.49	0.03	7.31	7.29	0.02
New Jersey (Rutgers)	C. S. Cathcart	4.47	4.45	0.02	7.30	7.23	0.07
New York (Geneva)	L. L. Van Slyke	4.49	4.45	0.04	7.33	7.27	0.06
New York (Geneva)	A. L. Knisely	4.46	4.42	0.03	7.27	7.19	0.08
North Carolina	F. B Carpenter	4.49	4.51*	0.02*	7.45	7.45	0.00
Pennsylvania	J. W. Fields	4.41	4.35	0.06	7.15	7.10	0.05
U.S. Bio-Chemical Laboratory.	J. A. Emery	4.52	4.56*	0.04*	.....	.....	.....
West Virginia	R. de Roode	4.47	4.47	0.00	7.32	7.26	0.06
Wisconsin	F. W. Woll	4.42	4.31	0.11	7.24	7.05	0.19
Average of all results		4.47	4.44	.....	7.27	7.21	.....
Difference of averages		.....	.....	0.03	.....	.....	0.06
Highest results		4.52	4.56	.....	7.45	7.45	.....
Lowest results		4.41	4.31	.....	7.15	7.05	.....
Difference between highest and lowest		0.11	0.25	.....	0.30	0.40	.....

* Increase by Gunning method.

STATEMENT OF RESULTS.

BONE MEAL:

(*a*) *Kjeldahl method.*—The extreme variation between the highest and lowest per cent. of nitrogen was 0.11 per cent, all results being within 0.06 per cent of the average.

(*b*) *Gunning method.*—The extreme variation between the highest per cent and the lowest was 0.25 per cent, all results except two being within 0.07 per cent of the average. It will be seen that the wider range by the Gunning method is caused by two of the thirteen results, one of which is 0.12 per cent above the general average and the other 0.13 per cent below the average. If we exclude these two results, the extreme variation between highest and lowest is 0.12 per cent, which is practically the same as the difference given by the Kjeldahl method.

Averaging all the results, the Kjeldahl method gave 0.03 per cent more of nitrogen than the Gunning. Two analysts obtained the same amount of nitrogen by both methods; two obtained 0.02 and 0.04 per cent more by the Gunning method; the others obtained from 0.02 to 0.06 per cent more by the Kjeldahl method except in a single instance, where 0.11 per cent was reported.

COTTON-SEED MEAL:

(*a*) *Kjeldahl method.*—The extreme variation between the highest and lowest result was 0.30 per cent, three results differing from the average by more than 0.10 per cent. Excluding these widest results, the extreme variation is 0.16 per cent.

(*b*) *Gunning method.*—The extreme variation was 0.40 per cent. Excluding three results which differed from the average by more than 0.10 per cent, the variation was 0.19 per cent, closely following the results of the Kjeldahl method.

Taking the average results the Kjeldahl method gave 0.06 per cent more than the Gunning. Two analysts reported the same results by the two methods; the others obtained from 0.02 to 0.08 per cent more of nitrogen by the Kjeldahl method, except one who reported 0.19 more.

As compared with the results reported by the Kjeldahl method last year, the results of this year are even better than the excellent results then reported, especially if the amount of nitrogen in the substances used is considered.

In reporting on the official samples, Mr. E. B. Voorhees says: "In 69 samples of commercial fertilizers, without nitrates, ranging in nitrogen from 0.85 to 8.51 per cent, the average analyses by association method, Kjeldahl, gave 2.394 per cent; by Gunning method, 2.407 per cent of nitrogen. Kjeldahl was higher than Gunning in 25 cases. The widest variation was 0.12 per cent in two cases. In 47 cases the difference was less than 0.05 per cent. The work was entirely satisfactory throughout, and I am satisfied that Gunning's method will supersede the Kjeldahl in this laboratory if further work verifies that already done."

Mr. C. S. Catheart report results of twelve analyses of milk by the Kjeldahl and Gunning methods, which can be summarized as follows:

	Per cent.
Average of results by Kjeldahl method	0.531
Average of results by Gunning method	0.528

In three cases the results were the same by both methods; in 6 cases the Kjeldahl gave from 0.01 to 0.02 per cent more nitrogen; in 3 cases the Gunning gave about 0.01 per cent more.

Omission of Permanganate in the Kjeldahl Method.

In the later reports of the association there occurs in the description of the Kjeldahl method the following sentence: "In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of this uncertainty it is always used."

The reporter on nitrogen last year made some study of this question and gave the conclusions derived from the results of his work in the following language: "The use of permanganate does not seem to have been of any benefit in the case of plain goods. The results are certainly sufficiently numerous and concordant to disprove the necessity for its use in the above samples. In the face, however, of strong statements in opposition to this conclusion, your reporter does not feel that it would be wise to change the official method at this point until a wider range of substances has been carefully studied. Nevertheless it should be noted that its use has already been discontinued by many of our chemists."

Omission of potassium permanganate in the Kjeldahl method.

State.	Analyst.	Bone meal.			Cotton-seed meal.		
		With permanganate.	Without permanganate.	Difference.	With permanganate.	Without permanganate.	Difference.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Arkansas	G. L. Teller	4.41	*4.42	*0.01	7.15	7.12	0.03
Illinois	E. H. Farrington	4.46	*4.48	*0.02	7.17	*7.23	*0.06
Kentucky	H. E. Curtis	4.50	4.48	0.02	7.27	7.16	0.11
Michigan (University)	E. R. Miller	4.44	*4.46	*0.02	.....	.....	.....
New York (Geneva)	L. L. Van Slyke	4.49	4.46	0.03	7.33	7.23	0.10
New York (Geneva)	A. L. Knisely	4.45	4.42	0.03	7.27	7.20	0.07
North Carolina	F. B. Carpenter	4.49	*4.53	*0.04	7.45	7.36	0.09
Pennsylvania	J. W. Fields	4.41	4.29	0.12	7.15	7.10	0.05
U. S. Bio-chemical Laboratory.	J. A. Emery	4.52	4.38	0.14	7.34	7.04	0.30
West Virginia	R. De Roode	4.47	*4.49	*0.02	7.32	7.24	0.08
Wisconsin	F. W. Woll	4.42	4.38	0.04	7.24	7.14	0.10
Average of all results		4.46	4.43	.....	7.27	7.18	.....
Difference of averages		.....	.....	0.03	.....	.....	0.09
Highest results		4.52	4.53	.....	7.45	7.36	.....
Lowest results		4.41	4.29	.....	7.15	7.04	.....
Difference between highest and lowest		0.11	0.24	.....	0.30	0.32	.....

*Increase without permanganate.

STATEMENT OF RESULTS.

BONE MEAL:

(*a*) *With permanganate.*—The extreme variation between the highest per cent and the lowest was 0.11 per cent.

(*b*) *Without permanganate.*—The extreme variation was 0.24 per cent. If the two widest results are omitted, the variation is 0.11, the same as in the preceding case.

The difference in the averages obtained with and without the use of permanganate is 0.03 per cent. In 5 cases, from 0.01 to 0.04 per cent more of nitrogen was given when no permanganate was used; in 4 cases the use of permanganate gave from 0.02 to 0.04 per cent more, while in 2 extreme cases the use of permanganate gave 0.12 and 0.14 per cent more. If we exclude these 2 cases and average the others we shall then obtain 0.01 per cent more with permanganate than without.

The results seem to indicate that in case of bone meal the use of permanganate is not necessary.

COTTON-SEED MEAL:

(*a*) *With permanganate.*—The variation between the extreme results was 0.30 per cent; excluding the highest and lowest results, the variation was 0.17 per cent.

(*b*) *Without permanganate.*—The extreme variation was 0.32 per cent; omitting, as above, the highest and lowest results, the variation was 0.14 per cent.

The difference in the averages of the two sets of results is 0.09 per cent. In only a single instance was more nitrogen obtained without permanganate; in all other cases, the use of permanganate gave from 0.08 to 0.11 per cent more than was obtained without permanganate; in one extreme case, 0.30 per cent more was given with permanganate.

The results seem to point to the conclusion that in case of cotton-seed meal the use of permanganate gives higher results by nearly 0.10 per cent, and that its use is necessary to insure complete conversion of organic nitrogen into ammonia.

Use of potassium permanganate in the Gunning method.

State.	Analyst.	Bone meal.			Cotton-seed meal.		
		With permanganate.	Without permanganate.	Difference.	With permanganate.	Without permanganate.	Difference.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent*
Arkansas	G. L. Teller	4.40	4.39	0.01	7.14	7.15	0.01
Illinois	E. H. Farrington	4.50	4.42	0.08	7.31	7.09	0.22
Kentucky	H. E. Curtis	4.45	*4.46	*0.01	7.18	*7.22	*0.04
Michigan (University)	E. R. Miller	4.40	*4.44	*0.04			
New York (Geneva)	L. L. Van Slyke	4.47	4.45	0.02	7.22	*7.27	*0.05
New York (Geneva)	A. L. Knisely	4.40	*4.42	*0.02	7.20	7.19	0.01
North Carolina	F. B. Carpenter	4.56	4.51	0.05	7.48	7.45	0.03
West Virginia	R. De Roode	4.48	4.47	0.01	7.29	7.26	0.03
Wisconsin	F. W. Woll	4.45	4.31	0.14	7.28	7.05	0.23
Average of all results		4.46	4.43		7.26	7.21	
Difference of averages				0.03			0.05
Highest results		4.56	4.51		7.48	7.45	
Lowest results		4.40	4.31		7.14	7.05	
Difference between highest and lowest		0.16	0.20		0.34	0.40	

*Increase without permanganate.

STATEMENT OF RESULTS.

BONE MEAL:

(*a*) *With permanganate.*—The variation between the highest and lowest results was 0.16 per cent, all results except one being within 0.06 of the average.

(*b*) *Without permanganate.*—The extreme variation was 0.20 per cent, all results except one being within 0.08 per cent of the average. Excluding the one result that was farthest from the average, the variation was 0.12 per cent.

The difference in the averages obtained with and without the use of permanganate is 0.03 per cent. In three cases, when no permanganate was used, from 0.01 to 0.04 per cent more of nitrogen was obtained; in five cases the use of permanganate gave from 0.01 to 0.08 per cent more, and in one case 0.14 more. If we exclude this one extreme case and average the others, then the averages obtained with and without permanganate differ by less than 0.01 per cent.

The results on the whole appear to indicate that the use of permanganate in the Gunning method in case of bone meal is not necessary.

COTTON-SEED MEAL:

(*a*) *With permanganate.*—The variation between the extreme results was 0.34, two results varying from the average by more than 0.10 per cent.

(*b*) *Without permanganate.*—The variation between extreme results was 0.40 per cent, three results not coming within 0.10 per cent of the average.

The average of the results with permanganate is 0.05 per cent higher than without permanganate; in three cases omission of permanganate gave from 0.01 to 0.05 per cent more; the difference in three other cases was from 0.01 to 0.03 per cent higher with permanganate; in two cases the difference was 0.22 and 0.23 per cent. Excluding these two results and averaging the others, we get the same average in both cases. It appears from these results that the use of permanganate is not necessary in the Gunning method in case of cotton-seed meal.

TEST OF ULSCH'S METHOD.

This method effects the reduction of nitrates to ammonia by means of reduced iron, as described on page 127, Bulletin 31.

Determination of nitrogen in nitrates by Ulsch and other methods.

State.	Analyst.	Sodium nitrate.			
		Ulsch method.	Kjeldahl-Scovell method.	Absolute method.	Ruffle method.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
New Jersey	J. P. Street	16.41	.....	.....	.....
Do	C. S. Cathcart	16.33	.....	.....	.....
New York	A. L. Knisely	16.17	.....	.....	.....
North Carolina	F. B. Carpenter	14.47	16.44	.....	.....
Pennsylvania	J. W. Fields	15.88	.....	.....	.....
U. S. Department of Agriculture	T. C. Trescot	16.34	16.36	16.40	16.33
Average of all results		15.93	16.40	16.40	16.33
Calculated per cent of nitrogen		16.49	.....	.....	.....
Highest result by the Ulsch method		16.41	.....	.....	.....
Lowest result by the Ulsch method		14.47	.....	.....	.....
Difference between highest and lowest		1.94	.....	.....	.....

Taking the calculated per cent of nitrogen as 16.49, three of the results come within 0.16 per cent of this amount, while three others vary from 0.32 to 2.02 per cent below the calculated amount. Mr. Carpenter reported six results, varying from 13.98 to 14.68 per cent. Mr. Knisely, in our Geneva laboratory, made eight determinations, with results varying from 16 to 16.25 per cent. It would appear that the method

tends to give low results in the hands of most of those who were not accustomed to using it. It is probable that with a more detailed description of the various steps of the process it might be made more reliable in the hands of all, but your reporter is unable to say just where the difficulty lies in the low results reported.

Summary of results furnished by study of methods.

BONE MEAL.

	Average of all results.	Average after excluding exceptional results.
Kjeldahl method (with permanganate)	4.47	4.47
Kjeldahl method (without permanganate)	4.43	4.45
Gunning method (without permanganate)	4.44	4.45
Gunning method (with permanganate)	4.46	4.46

COTTON-SEED MEAL.

Kjeldahl method (with permanganate)	7.27	7.27
Kjeldahl method (without permanganate)	7.18	7.20
Gunning method (without permanganate)	7.21	7.26
Gunning method (with permanganate)	7.26	7.25

COMPARISON OF STANDARD ACIDS.

A sealed bottle of hydrochloric acid was sent to each analyst with the request to compare with the standard acid of each and report results, as follows: (1) The amount of absolute hydrochloric acid in 1 cc. of analyst's acid, and (2) The number of cc. of analyst's standard acid that were found to be equivalent to 10 cc. of the reporter's standard acid.

The results reported are given below and are all expressed in terms of the reporter's acid, that acid being taken as 100:

	Per cent.
(1) New Jersey	99.5
(2) Pennsylvania	99.6
(3) U. S. Bio-Chemic Laboratory	99.7
(4) New Jersey (Rutgers)	99.8
(5) New York (Geneva)	100
(6) Kentucky	100.5
(7) Arkansas	100.7
(8) New Hampshire	101
(9) West Virginia	101
(10) U. S. Department of Agriculture	101.3
(11) Wisconsin	101.3
(12) North Carolina	101.4
(13) Illinois	102
Average of all	100.6

In examining the foregoing table we notice that there are four results between 99.5 and 100, and five of the others are between 101 and 101.4. Last year the results reported by nine analysts varied from 99.49 to 100.41, less than one-half the variation noticed this year. If the results of this year's work may be regarded as representing average work, the results of last year must be taken as of unusual excellence and not readily duplicated, since they vary rather less than duplicates are apt

to do in the hands of the same analyst working by one method and, certainly, less than results obtained by different methods of standardizing carried on in the hands of one analyst, as I will point out later.

Methods of Standardizing.

I will now present the reports of individual methods that were studied. I regret that there was insufficient coöperation in carrying out the work to the extent which was planned, and the results are far from exhaustive in any respect; however, as far as they go they are instructive and will prove of value.

STANDARDIZING BY SILVER CHLORID.

Gravimetric Method:

Mr. A. L. Knisely.—The hydrochloric acid was brought to the temperature of 16° and then 20 cc. were run out from an accurately graduated and calibrated burette, for each determination, into a weighing flask provided with a glass stopper. Different portions of 20 cc. run out in this way differed in weight by only a few milligrams at most. Below are given the results obtained in making repeated determinations by the silver chlorid method. Thefirst three determinations were made on the standard acid sent out this year, and the next set, consisting of four determinations, was made on another solution.

Mr. G. L. Teller.—The precipitated silver chlorid was collected on filter paper instead of using a Gooch crucible, for conveniences were not at hand for the latter. The individual results are not given but only the average of six determinations with maximum range, which is indicated below.

Mr. F. W. Woll.—Reports the individual results of six determinations by the official method.

In stating the results of the foregoing determinations, there is given (1) the number of grams of hydrochloric acid in 1 cc. of solution, expressed in terms of the reporter's acid, and (2) the amount of acid in each case, taking the reporter's acid as 100.

	Hydrochloric acid in 1 cc. of solution.	Compared with reporter's acid taken as 100.
	Grams.	*Per cent.*
Knisely	*a* 0.0083081	*a* 99.95
	b 0.0083167	*b* 100.06
	c 0.0083111	*c* 99.99
Average	0.008312	100.00
Extreme variation	0.0000086	0.11
Teller		*a* 100.02
		b 99.98
		c 99.73
		d 100.27
Extreme variation		0.54
Average	0.008371	100.7
Woll	*a* 0.0083483	*a* 100.44
	b 0.0083226	*b* 100.13
	c 0.0084190	*c* 101.29
	d 0.0083676	*d* 100.67
	e 0.0082453	*e* 99.20
	f 0.0083226	*f* 100.13
Average	0.0083376	100.31
Extreme variation	0.0001735	2.09

An examination of these results shows a greater variation in the work of 1 analyst than appeared in all the results reported by 9 different individuals last year.

VOLUMETRIC (silver chlorid):

Mr. G. L. Teller.—The silver nitrate solution was standardized by weighing the dry salt and checked by carefully prepared solution of sodium chlorid. Before titrating, the acid was neutralized by adding the exact amount of standard ammonia solution. The results obtained by the volumetric determination agree quite closely with those given by the gravimetric. Calling the gravimetric results 100, the volumetric results are expressed by the number 100.25.

Mr. F. W. Woll.—The average of 4 volumetric determinations gave 100.46 as compared with 100 by the gravimetric.

Standardizing by ammonium chloride.

	Hydrochloric acid in 1 cc. of solution.	Reporter's acid taken as 100.
Knisely	*a* 0.0085098	*a* 102.38
	b 0.0085098	*b* 102.38
	c 0.0083463	*c* 100.41
	d 0.0083833	*d* 100.86
	e 0.0083463	*e* 100.41
Average	0.0084191	101.29
Extreme variation	0.0001635	1.97
Woll	*a* 0.008843	*a* 106.39
	b 0.008547	*b* 102.83
	c 0.008425	*c* 101.36
	d 0.008605	*d* 103.52
Average	0.008605	103.52
Extreme variation	0.000418	5.03
Teller: Average of five determinations	0.008453	101.7

STANDARDIZING BY POTASSIUM TETRA-OXALATE.

To each analyst was sent a sample of tetra-oxalate, as obtained from Baker & Adamson, with the request to examine as follows: "Make a solution of definite strength, approximately decinormal and titrate (1) against 25 cc. of standardized solution of ammonium hydroxid, using for indicator litmus prepared according to diractions given in Sutton's Volumetric Analysis. For comparison of indicators use also dimethylanilin orange and cochineal solutions. (2) Titrate tetra-oxalate against 25 cc. of a standardized solution of sodium hydroxid, using as indicators, for comparison, solutions of phenolphthalein and cochineal. Recrystallize the tetra-oxalate three times and titrate as before, using the indicator that was found to give best results."

Taking the reporter's acid as 100, we give below the results obtained by each analyst in titrating the tetra-oxalate sent out against ammonia and fixed alkalis with the indicators mentioned:

Analyst.	Ammonia.			Sodium hydroxid phenolphthalein.	Barium hydroxid phenolphthalein.
	Litmus.	Cochineal.	Dimethyl anilin orange.		
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Jenter	89.10	90.05	Worthless.	90.48	.....
Parsons	90.60	.....	....do	.....	90.20
Fields	89.60	90.60	....do	.....	.....
De Roode	.....	.....	.....	91.30	.....

[Below are given the results obtained on the sample of tetra-oxalate after recrystallizing three times.]

Jenter	98.10	99.10	.....	{ 99.10 *99.97	.....
Parsons	101.38	.....	.....	.....	101.50
Fields	98.00	98.40	.....	101.60	.....
De Roode	.....	.....	.....	100.50	.....
Teller	99.89	.....	.....	.....	.....

* At the suggestion of Mr. Parsons I had some of the tetra-oxalate recrystallized with an excess of oxalic acid and below are the results obtained by Mr. Jenter with the different recrystallizations, using sodium hydroxide and phenolphthalein.

	Per cent.
Sample in original condition	90.48
Sample after one recrystallization:	
Without oxalic acid	95.00
With oxalic acid, in excess	96.90
Sample after two recrystallizations:	
Without oxalic acid	99.10
With oxalic acid, in excess	99.94
Sample after three recrystallizations:	
Without oxalic acid	99.10
With oxalic acid, in excess	99.97
A sample of tetra-oxalate from Eimer Amend gave	99.10

SUMMARY OF RESULTS FURNISHED BY STUDY OF METHODS OF STANDARDIZING.

In the table below are presented the results of standardizing by the various methods reported.

	Silver chlorid, gravimetric.	Silver chlorid, volumetric.	Ammonium chlorid.	Potassium tetra-oxalate.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Knisely (New York)	100.00	.....	101.29	.....
Jenter (New York)	.....	.....	.....	{ [1]99.10 [2]99.97
Teller (Arkansas)	100.70	100.90	101.70	99.89
Parsons (New Hampshire)	101.00	.....	101.10	{ [3]101.38 [4]101.59
Fields (Pennsylvania)	.....	.....	.....	{ [3]98.00 [1]101.60
De Roode (West Virginia)	.....	.....	.....	[1]100.5
Woll (Wisconsin)	100.31	100.77	103,52	.....

[1] Recrystallized three times without oxalic acid; titrated against sodium hydroxid with phenolphthalein as indicator.

[2] Recrystallized three times with excess of oxalic acid and titrated as above.

[3] Titrated against ammonia with litmus as indicator.

[4] Titrated against barium hydroxid with phenolphthalein.

We notice the following points:

(1) That the gravimetric determinations by silver chlorid are a little less than the volumetric determinations by silver chlorid.

(2) That the ammonium-chlorid determinations give in most cases higher results than any other method.

(3) That the determinations by potassium tetra-oxalate give lowest results when titrated against ammonia with litmus as indicator; highest results with sodium or barium hydroxid and phenolphthalein; and intermediate results with ammonia and cochineal.

Use of Indicators.

Each analyst was asked to state which indicator gave the most satisfactory results with potassium tetra-oxalate, and, in addition, to state which indicator, in his general experience, has been found to give the best results (1) in titrating inorganic acids against ammonia solutions; (2) in titrating inorganic acids against fixed alkali solutions; (3) in titrating organic acids against ammonia solutions; (4) in titrating organic acids against fixed-alkali solutions.

Jenter.—In titrating ammonia and tetra-oxalate, neither litmus nor cochineal gave sharp and satisfactory end reactions. Dimethylanilin orange was useless. The best results were obtained by titrating the tetra-oxalate against sodium hydroxid, with phenolphthalein as indicator.

Teller.—Using neutral litmus as indicator, no great difficulty was experienced in using ammonia with the tetra-oxalate, so far as distinguishing the end of the reaction is concerned. Cochineal was less satisfactory. Dimethylanilin orange was worthless.

Parsons.—With ammonia and tetra-oxalate, dimethylanilin orange and turmeric were found to be worthless. With litmus one or two drops were sufficient to change color, though it requires some practice to distinguish the final change sharply. Corallin gave a fairly sharp end reaction, but not so sharp as litmus. Phenolphthalein with barium hydroxid and tetra-oxalate gave sharp end reaction.

Fields.—Good results with litmus in case of tetra-oxalate and ammonia, but did not report in regard to distinctness of end reaction. Cochineal not as satisfactory as litmus. Dimethylanilin orange proved worthless. End reaction with phenolphthalein in case of tetra-oxalate and sodium hydroxid was satisfactory.

De Roode.—Sodium hydroxid and tetra-oxalate with phenolphthalein gave highly satisfactory results. Neither litmus, lacmoid, nor cochineal gave satisfactory indications with the tetra-oxalate solution with either ammonia or caustic soda. Lacmoid is better even than cochineal in titrating hydrochloric acid with ammonia.

The answers given to the general questions regarding the use of indicators may be summed up as follows:

(1) In titrating inorganic acids against ammonia there were given cochineal, litmus, and lacmoid. Cochineal has the advantage over litmus of keeping better.

(2) In titrating inorganic acids against fixed alkalis all agree on phenolphthalein.

(3) In titrating organic acids against ammonia solutions, litmus has the preference.

(4) In titrating organic acids against fixed-alkali solutions phenolphthalein is recommended.

Recommendations by the Reporter.

Your reporter recommends the following changes in the official methods for the determination of nitrogen:

First. On page 182, under "Apparatus and Reagents," change the last paragraph on the page so that it shall read as follows:

(1) Standard hydrochloric acid, the absolute strength of which has been determined by the following methods:

(*a*) By precipitation as silver chlorid. For this purpose bring the solution of hydrochloric acid to the temperature of 16° and then carefully run 20 cc. from an accurately graduated and calibrated burette, using a float to secure sharp readings, and then carry out the operation as described in Crookes' Select Methods, page 571.

(*b*) By potassium tetra-oxalate, which has been purified by recrystallizing three or four times with excess of oxalic acid. In titrating with this solution use standardized solution of sodium hydroxide and phenolphthalein as indicator.

(*c*) By ammonium chlorid, which must be chemically pure. A weighed quantity is boiled with an excess of sodium hydroxid and the distillate passed into a known volume of the hydrochloric acid.

(*d*) For ordinary work, half-normal acid, *i. e.*, acid containing 18.2285 grams of hydrochloric acid to the liter, is recommended; for work in determining very small amounts of nitrogen, one-tenth normal acid is recommended.

(*e*) In titrating hydrochloric acid and other mineral acids against ammonia solutions use cochineal as indicator.

Second. On page 183 change the first paragraph so that it shall read as follows:

(2) Standard ammonia, the strength of which, relative to the acid, has been accurately determined. One-tenth normal ammonia solution, *i. e.*, containing 1.7051 grams of ammonia to the liter, is recommended for accurate work.

Third. That the Gunning method as described below be made an official alternate method for determining nitrogen in substances which do not contain nitrates.

Fourth. On page 183, paragraph 11, add the following:

It is recommended that funnels having ground-glass stopcocks be used in the rubber stoppers of the distillation flasks and that the potassium sulphid and sodium hydroxid be added through these funnels after the flasks have been finally closed previous to distillation. It is also recommended that the end of the distillation tube dip under the surface of the standard acid into which the ammonia is received.

The president asked if there were any other papers on the subject.

Mr. FREAR. I have no paper to present, but there is one point that seems to me might be of interest in regard to the standard acid. The Pennsylvania results were 99.06 as compared with the rest. Last year it was 100. In most of the German stations they do not use permanganate, but they allow one hour for digestion, and I am anxious to know what the practice was in this case.

Mr. VAN SLYKE. There were no instructions on that point and no report made. We have digested until the solution was practically colorless or as nearly so as possible, running as our experience indicated a safe length of time, and if the solution fails to get colorless, the acid combustion runs about a certain time and is then stopped.

Mr. DE ROODE. I let the digestion flasks boil for at least three-fourths of an hour. I tried that in cotton-seed oil and I was as anxious to get as high a result without permanganate as with it, but I could not do it.

Mr. WILEY. It may seem like going back to old times, but I would like to report some results obtained by Mr. Trescot in our laboratory on these samples by two methods of which some of you have heard, viz, the absolute and the Ruffle.

The figures are are as follows:

	Per cent.
Absolute:	
No. 1	4.43
No. 2	7.52
No. 3	16.40
Ruffle:	
No. 1	4.51
No. 2	7.34
No. 3	16.33
Kjeldahl:	
No. 1	4.54
No. 2	7.34
Soda-lime:	
No. 1	4.37
No. 2	7.28

Mr. PARSONS. I have a paper on the subject that I would like to present.

COMPARISON OF METHODS FOR THE STANDARDIZATION OF ACID AND ALKALINE SOLUTIONS.

By E. M. PARSONS.

Owing to the recent interest in methods for the accurate determination of the strength of alkalimetric and acidimetric solutions, made manifest by numerous published papers and discussions, and to the undoubted importance of the subject, I have made many analyses by the more important methods in order to compare results.

Three solutions were prepared, one of hydrochloric acid about fifth-normal and one each of ammonia and barium hydroxid of approximately tenth-normal strength. These are the solutions commonly used in this country. They were made from pure chemicals and were protected from addition of moisture and of carbon dioxid where necessary.

The ammonia and barium hydroxid solutions were purposely made so that two cubic centimeters of either should exactly equal one of the hydrochloric acid.

The strength of the ammonia was determined directly by means of ammonium-platinum chlorid, by potassium tetra-oxalate, and by acid potassium tartrate. Portions of the barium hydroxid solution were precipitated as barium sulphate and were titrated against potassium tetra-oxalate, and the strength of the hydrochloric acid was ascertained by titration against ammonia, distilled from a known amount of ammonium chlorid; by pure, transparent, and colorless Iceland spar, and by precipitation as silver chlorid.

The remainder of the results given are, of course, obtained by comparison of the solutions, but are none the less important and accurate. Lack of time and, for the time being, inefficient batteries prevented my working with the copper-sulphate method of Hart and Croasdale, and I realize this to be a serious omission, which circumstances rendered unavoidable.

The indicators used were for each case those which in my experience have proved to be the best.

Standardization of HCl, NH_3, and $Ba(OH)_2$ solutions by different reagents.

1 cc. HCl sol. = 2 cc. NH_3 sol.

1 cc. HCl sol. = 2 cc. $Ba(OH)_2$ sol.

Method.	Indicator.	NH_3 in 1 cc. solution.	HCl in 1 cc. solution.	$Ba(OH)_2$ in 1 cc. solution.
		Grams.	*Grams.*	*Grams.*
By precipitation as $(NH_4)_2$ Pt Cl_6		.001545	.006634	
By neutralization with $KHC_2O_4H_2C_2O_4+2H_2O$	Litmus	.001560	.006698	
By neutralization with $KHC_4H_4O_6$	do	.001564	.006716	
By precipitation as AgCl		.001558	.006679	.007822
By pure pulverized Iceland spar	Cochineal	.001567	.006728	.007880
By distillation of NH_3 from NH_4Cl	do	.001563	.006717	.007866
By precipitation as $BaSO_4$			.006676	.007820
By $KHC_2O_4H_2C_2O_4+2H_2O$	Phenolphthalein		.006722	.007875

The potassium tetra-oxalate was prepared by myself from K_2CO_3, and $H_2C_2O_4$, and was recrystallized six times, being washed once with distilled water by decantation after each crystallization.

The acid potassium tartrate was prepared by myself from the purest cream of tartar obtainable, was heated with HCl, washed and crystallized according to directions of Dr. Bornträger (Zeit. für anal. Chem., Vol. 31, p. 56), and was further recrystalized three times, as the directions given did not prove sufficient in my hands to give a pure salt.

A glance at the above table indicates one general feature, which is, that in every case the figures obtained by gravimetric methods are lower than those obtained by the volumetric. This is probably what would be generally expected, for, of the two, the tendency toward loss of substance and consequently lower results is certainly more for methods involving the manipulation incident to gravimetric ones. The results are, however, on the whole, good. Those obtained by silver chlorid and barium sulphate agree closely, and the lower result obtained by ammonium-platinum chlorid is easily accounted for by the comparatively high solubility of the salt. I have no hesitancy, however, when convenience is also taken into consideration, in placing the determinations as silver chlorid at the head of gravimetric methods.

With volumetric methods the case is somewhat different. The absolute composition and purity of the salt used as a standard and its efflorescent and deliquescent properties become of the greatest importance. No substance whose accurate composition can not be definitely known at any time can be used. I will take up each method by itself.

By distillation of ammonia from an alkaline solution of ammonium chlorid results seem to be invariably high while still agreeing fairly well with other methods. This is the general testimony of chemists, and the reason may possibly be ascribed to a slight tendency of the alkali, used to decompose the chlorid, to pass over mechanically. But as the higher results are obtained after all precautions are taken these conclusions may be doubted.

The reaction obtained with cochineal is of course sharp and duplicates generally agree closely.

The Iceland spar used was transparent and colorless, was finely pulverized, and dried for several hours at 100°. The results here are also slightly high, but the tendency is also toward high results, not only on account of the danger of the presence of minute quantities of carbon dioxid not removed by boiling, but from the fact that the titration is carried from acid to alkaline reaction and calculations made directly on acid. The one or two extra drops of alkali required to show alkaline over acid color easily account for most of the increase. Unless the mineral is very finely pulverized it dissolves but slowly in the very weak acid.

The results obtained by acid potassium tartrate were near the average after the salt was obtained pure. The preparation of the salt is, however, tedious on account of its extreme insolubility, and the tendency is to be too acid and consequently to give low results. The chief difficulty encountered, however, was my inability to find an indicator except phenolphthalein whose reaction should be as definite as

desired in its presence. It was found necessary for accurate work to average several analyses. Four or five drops of decinormal ammonia solution were generally necessary to show certain color in either direction. The salt does not contain water of crystallization and may be dried at the temperature of boiling water.

The determinations made by the aid of potassium tetra-oxalate are almost exactly the average of all results, both gravimetric and volumetric, and are in my opinion the most accurate.

I shall attempt to show in the following paper that the salt is easily and definitely prepared and that the chief objection raised against it, viz, that it contains water of crystallization, is, in this case at least, inoperative. Its reaction with litmus prepared according to Sutton is decidedly sharp for an organic salt and most excellent duplicates are easily and quickly obtained. The fact that much of the tetra-oxalate on the market is a mixture of the tetra and normal oxalate, and that before recrystallization many have not first added excess of oxalic acid does, to my mind, account for the recent disrepute into which the salt has apparently fallen.

Potassium Tetra-Oxalates.

The following set of analyses was undertaken to find out if possible the reason why potassium tetra-oxalate has failed to give good results in the hands of many chemists as a standard for alkalimetric work, while it has been held by others to give perfect satisfaction.

In order to study commercial samples, an order was sent to six different firms for 1 ounce of their best tetra-oxalate, and part of the firms were asked to send 1 ounce of each make they had in stock, giving manufacturers' names. The results on the samples received, with a few samples from other sources, are given in the following table:

Potassium tetra-oxalates.

(Comparison of different makes, keeping qualities, hygroscopicity, recrystallization, etc. Litmus, having been found to be the best indicator, was used. Prepared according to Sutton.)

[1 cc. NH_3 sol. = .001558 by comparison with HCl by AgCl.]

No.	From whom received.	Manufacturer or name on label.	How prepared for analysis.	Amount of NH_3 in 1 cc. NH_3 solution.
1 *a*	Mr. Van Slyke	Baker & Adamson	As received	.001731
*2 *b*	do	do	Recrystallized 3 times. Dried over H_2SO_4.	.001569
†2 *a*	Eimer & Amend	Eimer & Amend	Recrystallized 3 times in September, 1889, with excess of oxalic acid. Not dried for 2 years 8 months.	.001574
2 *b*	do	do	Same, but dried 18 hours over H_2SO_4.	.001575
3 *a*	do	Made in our laboratory from K_2CO_3 and $H_2C_2O_4$.	Recrystallized 3 times. Dried over H_2SO_4.	.001565
3 *b*	do	do	Recrystallized 6 times. Dried over H_2SO_4.	.001561
4	Eimer & Amend	Eimer & Amend	As received, but dried over H_2SO_4.	.001568
5	Richards & Co	E. Merck	do	.001572
6	Landenberger	do	do	.001571
7	E. H. Sargent & Co	E. H. Sargent & Co	do	.001858
8	J. W. Queen & Co	Baker & Adamson	do	.001720
9	do	E. Merck	do	.001563
10	Henry Heil & Co	do	do	.001574

*Excess of oxalic acid added before a recrystallization.

†Was ordered as tetraoxalate and so labeled, but was barely acid in reaction; was nearly the normal oxalate.

CHARACTER OF SAMPLES ON THE MARKET.

An examination of the above figures shows at once that while potassium tetra-oxalate is obtainable of a fair degree of purity the chemical market can not in this case, as in many others, be depended upon to furnish a strictly chemically pure salt. In discussing the subject I shall take the result under 3 *b* as a standard, believing it to be more accurate than that obtained by comparison with the hydrochloric-acid solution standardized by silver chlorid, although this result is placed above the table that any may refer to it who desire.

Leaving out No. 2, which was ordered in September, 1889, and which, when received, turned out to be the almost pure normal oxalate, we find that three, viz, Nos. 1 *a*, 7, and 8, were mixtures of acid potassium oxalate and the tetra-oxalate, while the other five, four of which were of Merck's guaranteed manufacture, were practically pure. It is especially noticeable that in no case was there an excess of oxalic acid present, while in Nos. 1 *a*, 7, and 8 a sufficient quantity had not been added. Nos. 4, 5, 6, 9, and 10 run quite close together, and are nearly up to the standard of a tetra-oxalate recrystallized three times after addition of oxalic acid, but each might be somewhat improved by such recrystallization.

PREPARATION OF PURE POTASSIUM TETRA-OXALATE.

This is easily and speedily accomplished either from the tetra-oxalates as bought or from potassium carbonate and oxalic acid. In the cases 1 *b*, 2 *a*, 2 *b*, 3 *a*, and 3 *b* the salt was recrystallized from boiling solution with addition of oxalic acid previous to the first crystallization. In each case the salt came down in well-formed crystals on the natural slow cooling of the solution, the supernatant liquid was discarded, the crystals were washed once by decantation with distilled water, recrystallized again from pure distilled water a sufficient number of times, pressed between filter papers and dried over night in a desiccator above sulphuric acid. It seems to be advisable to continue the first heating in presence of oxalic acid for at least an hour. This was specially apparent in the case of 3 *a*. Sufficient oxalic acid had been added, but the crystals first formed were a mixture similar to Nos. 1 *a*, 7, and 8, and were only purified on further addition and heating with oxalic acid. The last part of the reaction seems to take place slowly, which may account for the trouble in the three impure samples. The work was very easily carried out, three crystallizations being easily made in the course of as many hours with little time taken from other work. In no case was there an excess of acidity or a loss of water of crystallization, either of which would mean a decrease in the amount of ammonia found in one cubic centimeter. The fact that this is the case and that the results compare as they do with those obtained by other methods of standardization is largely proof of the purity of the salt. If there had been an excess of acidity, as claimed by many members of last year's Association of Official Agricultural Chemists, the results would be lower than those obtained by silver chlorid, for more acid to a certain weight means that a greater number of cubic centimeters of the ammonia solution would be required to neutralize that acid, and consequently one cubic centimeter on calculation would appear to contain less ammonia. This is contrary to the general consensus of opinion that potassium tetra-oxalate gives higher results than silver chlorid, and taken in connection with the above table of results leads me to believe that excess of oxalic acid is never a fault in a properly recrystallized or a commercial chemically pure potassium tetra-oxalate. As the recrystallization of the salt is so easily accomplished, I believe it best, for the sake of extreme accuracy, to recrystallize more than three times, and the results in 3 *b* would seem to indicate this. While the change made by the three extra crystallizations is only slight, I believe it to be worth while to do this extra work as a safeguard, especially as the salt once prepared will be of service for a long time.

DOES THE SALT LOSE OR GAIN WATER UNDER ORDINARY CONDITIONS?

Potassium tetra-oxalate loses water of crystallization at 100° quite noticeably, and drying at this temperature is out of the question. Such drying, however, is by no means necessary, or even desirable. Fortunately, for the purposes of this section, I had prepared some tetra-oxalate from normal potassium oxalate nearly three years ago. During this time the bottle containing the salt has been opened from time to time and portions removed, but the remainder had not been dried or otherwise tampered with during that time. It gave as removed from the bottle without drying a result, 2 *a*, fully as close to that obtained by silver chlorid as when the salt was first prepared, fairly within the limits of error, and agreeing closely with the other freshly prepared and three-time recrystallized tetra-oxalates. This would show that the substance was not hydroscopic, as it had been freely exposed to atmospheric influences. The result given in 2 *b*, reached after drying the same crystals for eighteen hours over sulphuric acid shows that no loss of water of crystallization took place during three years. In order to test this latter point still further, I placed 6 grams of broken fragments of crystals of potassium tetra-oxalate in a desiccator over fresh concentrated sulphuric acid. For several days there was no loss apparent to the balances, but after forty days a loss of one milligram was indicated. This loss when calculated to the 2 grams tetra-oxalate, used in an analysis, amounts to .00032 gram, a quantity much less than any of our balances will show. The loss was now more rapid, but even when the last weight was taken after three months' drying the loss was insignificant. It was sufficient merely to change the result, .001561 to .00156097, just three points in the eight-decimal place. As it is never necessary to dry the tetra-oxalate over sulphuric for more than twelve hours, I think I can safely say that the presence of water of crystallization in this salt is in nowise an objection.

Phenolphthalein, litmus, and corallin give a sharp reaction, but phenolphthalein is to be recommended where fixed alkali solutions are to be titrated against.

Litmus prepared according to Sutton gives a sharp reaction with ammonia solution. The change of color should be from acid to alkali and the point taken the distinct appearance of the blue. The shade of red begins to change before this point is reached, but the appearance of the blue is sharp and not to be mistaken. Well-agreeing results can be obtained with varying quantities of tetra-oxalate, which would not be the case if the change were doubtful. Corallin also has a fairly sharp reaction, but does not equal litmus in this respect.

Cochineal becomes decidedly alkaline in color several tenths of a cubic centimeter before this point is reached with any of the preceding and can not be used.

Dimethyl orange changes gradually during the addition of many cubic centimeters of alkali and first becomes fully alkaline in color when the tetra-oxalate is about half neutralized. As has long been known, this indicator can not be used with organic acids, nor does this case prove to be an exception.

H. Bornträger (Chemiker Zeitung, 1881, p. 519), H. Heidenhain (Zeit. für anal. Chem., 1891, p. 226), and A. Bornträger (Zeit. für anal. Chem., 1892, p. 56), have recommended acid potassium tartrate as a means for the standardization of acid and alkaline solutions. In order to try this salt I made up some according to the directions given by the last-named person, but found it necessary, in order to get the pure salt, to crystallize three times in addition.

The following are the results obtained:

	Grams NH_3.
Prepared according to direction 1 cc. NH_3 =	.001536
Recrystallized twice ..... 1 cc. NH_3 =	.001551
Recrystallized three times ..... 1 cc. NH_3 =	.001564
By potassium tetra-oxalate	.001561

Good results were obtained with the pure salt. The reaction with litmus was, however, not so sharp as with tetra-oxalate, and a number of determinations must be made with this indicator to be sure of results. It has also the disadvantage of

being much more tedious to prepare than the tetra-oxalate, and, so far as my work goes, shows no advantages whatever over the latter.

The tendency in the above is to retain an excess of acid, probably tartaric, set free in small quantity by the hot hydrochloric acid. The acid tartrate was washed after boiling with hydrochloric, until the latter had been completely removed.

Mr. De Roode. I have some statistics on the subject. They were made on different substances. The analyses were made without permanganate in the first and with it in the second column. I never have had any fertilizers in our State that contain cotton-seed meal.

Nitrogen determinations.

Without $KMnO_4$.	With $KMnO_4$.	Without $KMnO_4$.	With $KMnO_4$.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
.39	.36	2.02	2.03
.60	.60	2.11	2.10
.60	.60	2.20	2.26
.61	.60	2.22	2.22
.93	.92	2.28	2.22
1.03	1.06	2.35	2.30
1.07	1.07	2.34	2.28
1.20	1.16	2.42	2.46
1.21	1.18	2.48	2.48
1.25	1.21	2.54	2.55
1.31	1.30	2.55	2.51
1.34	1.30	2.65	2.64
1.36	1.36	2.73	2.70
1.38	1.37	2.90	2.89
1.39	1.38	2.94	2.95
1.45	1.45	3.02	3.04
1.51	1.53	3.11	3.16
1.56	1.59	3.36	3.38
1.64	1.61	3.75	3.71
1.83	1.80	3.77	3.79
1.92	1.87	3.88	3.91
1.97	1.95	4.06	4.08
1.98	1.98	4.49	4.44
2.03	1.98	4.49	4.49
2.05	1.99	4.54	4.54
2.02	2.02	7.24	7.32
2.08	2.02	7.23	7.31

The President. We are not half through yet and we will have to condense our discussion if we are to get through here to-morrow.

Mr. Voorhees. I have a paper here that I think is hardly necessary to read because I have the results tabulated; I submit it as it is.

THE GUNNING-KJELDAHL METHOD AND A MODIFICATION APPLICABLE IN THE PRESENCE OF NITRATES.

By E. B. Voorhees.

The Gunning-Kjeldahl method for the determination of nitrogen in fertilizers free from nitrates, suggested by the reporter on nitrogen, was used against the Kjeldahl method in the laboratory of the New Jersey Station the past season. The results

secured were very satisfactory, and the method much preferred to the Kjeldahl, mainly because it obviates the use of potassium sulphid, which is a disagreeable reagent to handle, besides adding weight to the solution.

A study of the tabulated analyses of 108 samples which were determined by the two methods shows that the range in total nitrogen is from 0.61 per cent to 8.51 per cent. The average of the whole number of determinations is 2.38 per cent nitrogen by the Kjeldahl method, and 2.397 per cent nitrogen by the Gunning-Kjeldahl, a difference of but .017 per cent nitrogen. The greatest difference in any case is 0.12 per cent and the average difference is .047 per cent.

In 59 cases the Gunning method gave the higher result; in 41 cases the lower, and in 8 cases the results were identical. No difficulty was experienced in the use of the method from the beginning though the digestion seems to be less rapid than in the Kjeldahl, because, owing to the frothing, it must proceed more slowly at first.

A MODIFICATION OF SCOVELL-JODLBAUR, APPLICABLE IN THE PRESENCE OF NITRATES.

In the determination of nitrogen in fertilizers containing nitrates, 10 grams of sulphate of potash was substituted for the metallic mercury of the official method. The method used was as follows: About 1 gram of the sample is weighed into a round-bottomed flask of 500-600 cc. capacity, suitable for both digestion and distillation, 10 grams of potassium sulphate are then added, then 30–35 cc. of the salicylic acid mixture (30–35 cc. of H_2SO_4 and 1 gram of salicylic acid), and 3 grams of zinc sulphid. The substance is then digested at a low temperature until frothing ceases, and then strongly until the solution is colorless, or nearly so; the whole time of digestion is usually from two to three hours. Before the solution is cool it is diluted with about 200 cc. of water, and the determination finished as in the Gunning-Kjeldahl method.

The use of this method was begun early in the season, and the results compare so favorably with those obtained by the official method that it was used as a check in all samples containing nitrates. The only precaution necessary to be observed is to digest at a low temperature at the beginning, and then to continue the strong digestion for at least two hours. The advantage of this method over others that have been suggested is mainly due to the fact that all the necessary reagents may be added at once and in the beginning of the determination.

The results secured without any attempt to adapt the method to other than regular laboratory work are as follows: In the 55 samples analyzed the total nitrogen ranged from 1.35 per cent to 11.43 per cent. These samples contained nitrate nitrogen ranging from 0.10 per cent to 5.52 per cent; they also contained widely varying amounts of nitrogen as ammonia, and of phosphoric acid and potash, as would be expected from so large a number of different brands of commercial fertilizers. The average of the whole number of determinations by the official method is 3.48 per cent, and by the modified method 3.47 per cent. The greatest difference in any case is 0.12 of one per cent; the average difference is 0.05 of one per cent. In 36 samples the modified method gave the lower result, in 18 samples the higher, and in one the results were identical.

Nitrogen determinations in fertilizers free from nitrates.

[By the Kjeldahl method and by Gunning-Kjeldahl method.]

Kjeldahl method.	Gunning-Kjeldahl method.	Kjeldahl method.	Gunning-Kjeldahl method.	Kjeldahl method.	Gunning-Kjeldahl method.	Kjeldahl method.	Gunning-Kjeldahl method.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
3.01	3.04	0.94	0.89	1.39	1.48	2.25	2.24
1.31	1.33	3.36	3.24	2.76	2.82	4.01	4.08
2.33	2.32	1.59	1.60	2.48	2.59	2.66	2.62
8.46	8.51	1.74	1.77	3.94	3.90	1.88	1.78
2.71	2.70	2.66	2.69	2.90	2.89	1.05	1.05
4.09	4.06	2.45	2.46	2.03	2.06	2.35	2.44
2.75	2.74	0.85	0.96	2.66	2.77	1.52	1.53
2.52	2.62	2.23	2.23	3.19	3.15	2.41	2.43
2.36	2.42	1.93	1.97	2.34	2.39	2.32	2.29
3.74	3.81	1.53	1.50	2.54	2.47	2.38	2.46
4.86	4.94	1.72	1.77	2.40	2.50	1.79	1.73
1.30	1.28	2.69	2.74	2.54	2.57	2.75	2.71
1.82	1.89	3.04	3.02	0.61	0.60	0.79	0.88
1.37	1.37	2.07	2.07	2.57	2.25	3.52	3.56
1.94	1.89	1.99	2.02	1.16	1.15	6.10	6.23
1.96	1.99	2.22	2.31	1.76	1.75	2.62	2.71
2.19	2.14	2.26	2.32	3.27	3.27	3.68	3.62
3.68	3.67	3.25	3.25	1.57	1.58	2.62	2.69
3.15	3.12	3.24	3.33	1.76	1.77	0.73	0.73
1.84	1.82	0.93	0.94	1.63	1.59	2.17	2.26
1.12	1.01	1.31	1.28	2.07	2.19	1.42	1.46
1.31	1.31	3.01	3.08	2.66	2.69	2.76	2.85
2.54	2.42	1.31	1.38	3.81	3.78	2.40	2.39
2.00	2.01	2.04	2.09	1.70	1.69	3.33	3.35
2.82	2.85	4.51	4.53	1.24	1.33	1.95	1.91
1.64	1.71	1.03	1.06	1.94	1.85	0.42	0.48
0.86	0.83	2.55	2.59	4.21	4.17	3.93	4.01

Nitrogen determinations in fertilizers containing nitrates.

[By the Scovell-Jodlbaur method and by Gunning's modification of the same.]

Scovell-Jodlbaur.	Gunning-modification.	Scovell-Jodlbaur.	Gunning-modification.	Scovell-Jodlbaur.	Gunning-modification.	Scovell-Jodlbaur.	Gunning-modification.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
3.12	3.08	1.52	1.47	3.70	3.61	4.49	4.48
3.52	3.57	3.44	3.42	3.28	3.27	2.18	2.11
4.00	3.95	1.93	1.95	3.76	3.73	4.13	4.06
2.23	2.23	3.50	3.39	2.04	2.09	3.31	3.28
4.22	4.30	2.99	2.88	3.11	2.99	4.08	3.99
3.04	3.05	1.35	1.38	2.66	2.64	2.37	2.46
1.18	1.15	3.14	3.26	4.19	4.29	1.88	1.88
4.12	4.11	3.12	3.05	2.64	2.55	3.09	3.01
3.28	3.18	2.84	2.82	4.43	4.44	10.18	10.24
2.63	2.74	4.21	4.10	2.99	2.89	11.35	11.43
2.94	2.88	3.42	3.47	3.37	3.38	7.88	7.78
2.73	2.71	1.78	1.71	3.28	3.19	2.63	2.71
2.86	2.85	2.62	2.61	3.28	3.31	3.87	3.97
4.30	4.40	4.19	4.16	3.40	3.31		

The following paper on the subject under discussion was read:

THE ULSCH METHOD IN DETERMINING NITRIC NITROGEN IN COMPLETE FERTILIZERS.

By JOHN PHILLIPS STREET.

The estimation of nitric nitrogen by reduction with iron, as suggested by Ulsch (Zeitschr. anal. Chem., 1891, 30, 175–182), possesses two important advantages; it is accurate and it is rapid. While the Schultze-Tiemann method for nitrates, has given satisfaction as a rule, yet the fact that it is a slow method militates strongly against its use where any large number of analyses must be made.

The Ulsch method as suggested applies only to nitrate salts, and for this purpose it has been found to be very satisfactory. A sample of pure sodium nitrate was analyzed, both by the Schultze and the Ulsch method, with the following results:

Method.	No. 1.	No. 2.	No. 3.	Average.
Schultze	16.39—	16.44—	16.45	16.43
Ulsch	16.44—	16.37	.....	16.41
Theory	.....	.....	.....	16.47

This application of the method proving satisfactory, it was then decided to test its accuracy when determining the nitrates in a complete fertilizer. The percentage obtained would, of course, represent not only the nitrogen as nitrates, but in addition to this the nitrogen from ammonia salts. Several samples were examined regarding this point. The method outlined in the Proceedings of the Association of Official Agricultural Chemists, 1891, p. 127, was followed closely, and in every case the results were high. This disparity suggested the idea that perhaps the caustic soda had liberated some of the organic nitrogen present and thus caused high results. The same samples were then analyzed again, using the same method, with the exception that instead of caustic soda heavy magnesia was used. Smaller amounts of iron, namely, 2 and 1 grams, were also used.

The results obtained by these various tests are here tabulated:

Sample No.	By present methods.				By Ulsch methods.			
	Nitrate.	Ammonia.	Organic nitrogen.	Nitrates plus ammonia.	Caustic soda, 5 grams iron.	Caustic soda, 2 grams iron.	Magnesia 2 grams iron.	Magnesia 1 gram iron.
104	0.66	1.05	2.26	1.71	1.97	1.85	1.73	1.72
158	1.74	2.03	.70	3.77	3.97	3.91	3.73	3.69
239	1.16	.68	2.45	1.84	2.16	2.33	1.81	1.81
241	.86	1.35	2.23	2.21	2.46	2.37	2.16	2.10

It will be seen from these figures that in two of the four samples the results from using magnesia were practically identical with the theoretical composition; that in the other two, while somewhat wider, the greatest variation was 0.11 per cent, and that in every case the caustic soda gave much higher results, ranging from 0.14 per cent to 0.49 per cent. The quantity of iron used did not seem to affect the results, just as good results being obtained from the use of 1 gram as from 5.

It will be noticed that all the samples were fairly high in nitrogen, ranging from 3.97 per cent to 4.51 per cent, and also that they all contained proportionately large amounts of nitric, ammonia, and organic nitrogen.

The average results show the relative effects of the caustic soda and the magnesia still more strikingly.

	Per cent.
Theory	2.38
Caustic soda and 5 grams iron	2.64
Caustic soda and 2 grams iron	2.62
Magnesia and 2 grams iron	2.36
Magnesia and 1 gram iron	2.33

Method.—The method changed in accordance with the suggestions given above would read as follows:

Weigh off 1 gram of sample into a 500 cc. flat-bottomed flask. Add about 30 cc. of water, 1 gram of reduced iron and 10 cc. of sulphuric acid (1—1). Shake well and allow to stand for a short time. This will remove the danger of an explosion caused by the otherwise violent action which takes place. Close the neck of the flask with a rubber stopper through which passes a glass dropping bulb filled with water. The flask having been stoppered, place it on a soapstone griddle to which a moderate heat is applied. Allow the solution to come slowly to a boil and then boil for five minutes. Cool. Add about 100 cc. of water, a little paraffin and about 5 grams of magnesium oxid. Boil for forty minutes, after which time all the ammonia will be distilled off.

Cautions.—The caustic soda causes a very bad frothing, which it is almost impossible to overcome. The magnesia causes a slight frothing, which can be easily controlled by adding a little paraffin and by bringing to a boil very gradually. Fully forty minutes are necessary to distill all the ammonia. Tests were made after thirty minutes' boiling and traces of ammonia were still found; after forty minutes these traces entirely disappeared.

Observations.—(1) The method is a quick one. One man can easily do six determinations at a time, and these six determinations can be made in but a little over an hour. (2) Caustic soda gives high results and is almost useless because of the great amount of frothing it occasions. (3) Magnesia gives results closely agreeing with theory and causes a very slight frothing, which can be easily controlled. (4) One gram of iron is sufficient in all ordinary complete fertilizers.

There being no further papers, the president called for discussion of the methods.

Mr. SCOVELL. I have one or two points to which I wish to call attention on the use of potassium sulphate in place of mercuric oxid in determining the nitrogen in the form of nitrates.

Results on potassium nitrate.

	Scovell-Kjeldahl.	Same except using K_2SO_4.
	Per cent.	*Per cent.*
1	13.74	13.72
2	13.75	13.74

I believe the results are highly satisfactory.

The PRESIDENT. We are prepared for a discussion of the recommendations now.

Mr. VAN SLYKE read the first recommendation.

Mr. WHEELER. I would like to say that in our laboratory in the summer the temperature ranges anywhere from 22° to 28°, and the

solution used is measured off at those various temperatures, and it seems to me this is a matter where we might use a little discretion—individual discretion—according to the circumstances and the season of the year.

Mr. PARSONS. We should have the laboratory under ordinary working conditions. The new kind of burette with the enameled back showing the point of light is, it seems to me, much better than a float.

Mr. WOODS. There is in my judgment too much detail in the description of the apparatus to be used. I think that in our official methods it is a little dangerous to do too much of that. The essential point in all our work is accurate measurements in accredited apparatus. One man uses a float and another an enameled burette. The man that has used the float will make a botch of it if he tries to use the burette, and *vice versa.* I think we should respect those peculiarities. I do not believe we can make chemists by making our rules too binding on them, and I would like to see the details left to the judgment of the chemists.

The PRESIDENT. Does the first recommendation meet with a second?

Mr. VAN SLYKE. I withdraw it.

The PRESIDENT (to Mr. Van Slyke). Give us the second recommendation.

Mr. VAN SLYKE read the second recommendation and then said: These recommendations are presented simply for consideration and with the expectation that they will be dealt with according as thought best.

Mr. DE ROODE. I move that that be left out entirely.

Seconded.

The PRESIDENT. Do you, Mr. Van Slyke, accept that?

Mr. VAN SLYKE. I had just as lief withdraw that.

Mr. DE ROODE. I move to strike out the whole thing in regard to the tetra-oxalate method.

Seconded.

Mr. VAN SLYKE. My feeling is that we have the result of only three or four workers on the matter, and that we should have more. I should like to see more work on it.

The PRESIDENT. I suggest that we leave it alone until another year and call the attention of the reporter to it next year.

Mr. WINTON. It seems to me that we should not adopt it without further trial.

Mr. DE ROODE. I move that this be stricken out and that the matter be referred to the reporter for further consideration next year.

Seconded.

Mr. VAN SLYKE. Is it intended to leave the method in there as it was last year.

The PRESIDENT. No; to strike it out entirely.

It was stricken out by a vote of 9 to 7.

Mr. VAN SLYKE read the next recommendation and said: I suspect the difficulty has come from using impure ammonium chlorid, because we base our determinations on absolutely pure ammonium chlorid.

Mr. WOODS. You say pure ammonium chlorid. Ought not the phraseology to be changed?

Mr. VAN SLYKE. This goes a little more into detail. I have known young men who have been away at stations, who have not attended our meetings and talked with our chemists and taken our reports, and with the meager descriptions given have not known exactly how to go to work.

Mr. ROSS. In regard to the use of ammonium chlorid would it not be well to try some ammonium salt containing a small relative proportion of ammonium?

Mr. SCOVELL. I do not see the necessity of using ammonium chlorid when we can not get an accurate result. I move the recommendation for its use be stricken out.

Mr. LORD. That will leave us only one way of standardizing our acid.

Mr. WILEY. That is the best condition to be in. It is not safe to measure by two standards.

Mr. VAN SLYKE. If we strike out the tetra-oxalate method, which is found to give better results than the ammonium chlorid, we will be left in a rather illogical condition of affairs if we retain the ammonium chlorid.

Mr. SCOVELL'S motion was carried.

Mr. DE ROODE. My substitute which I offer is an alternative method for standardization, the proportion of the solution of sulphuric acid to be one-fourth normal and the determination of the strength of the acid by means of substitution of barium chlorid. This is a method that I think all of you will have great confidence in. I place more confidence in this than in silver chlorid. By using them both and taking an average I think I can get the correct mean.

The PRESIDENT. Give your resolution in full.

Mr. DE ROODE. I move an alternative method for standardizing the acid, that a solution of diluted sulphuric acid approximately one fourth normal be made, and then the strength of this acid accurately determined.

Mr. WILEY. If the gentleman will allow me to interrupt him, I wish to say that I think it is not well to adopt any method for an alternative method until investigated by the reporter. I think that is a rule we are now coming to, and a very good one, and if Mr. de Roode change his motion so as to request the reporter to investigate the matter in the coming year I think it would be better.

Mr. DE ROODE. I am willing to change it that way.

The PRESIDENT. That is a good suggestion, as I think we ought to be careful about changing our methods.

The motion was carried that the subject be recommended to the reporter for next year.

Mr. VAN SLYKE. The next recommendation is simply changing the factor.

Carried.

The recommendation was seconded and adopted.

The next recommendation as to changing the wording of the first paragraph on page 183, Bull. 31, was read.

Adopted.

The next recommendation (3) was read.

Adopted.

Mr. VAN SLYKE. Here is one in the nature of the first one. It is gratuitous on my part. On page 183, paragraph 11, add the following: "It is recommended that funnels having ground-glass stopcocks be used in the rubber stoppers of the distillation flasks, and that the potassium sulphid and sodium hydroxid be added after the flasks have been finally closed previous to the distillation. It is also recommended that the end of the tube dip under the surface of the standard acid into which the ammonia is received." Perhaps there is not any loss as a rule, and perhaps there is, but it would be well to be on the safe side, and nobody is obliged to use it if he doesn't want to.

Mr. WHEELER. I have known of loss in such cases where the solution was cooled off previous to the addition of the suphid. I have adopted the custom, when they are warm, of putting them in a water bath and cooling them before it was added.

Mr. SCOVELL. I object to the form of the flask. I think we get as accurate results without the stopcock.

Mr. VAN SLYKE. This was a mere suggestion. Prof. Harrington was at our station last summer, and he, in looking around, said he used it in his laboratory and thought it was very desirable. I withdraw the first portion of this in regard to the funnel business and present only the last portion.

Mr. DE ROODE. I would like to amend it so as to read "until about 100 cc. are distilled over."

Mr. VAN SLYKE. It seems to me it is not necessary to go into such details.

Mr. DE ROODE. If you do not go into detail then you let the chemist use his judgment about it.

Mr. WILEY. That is the best way.

Mr. VAN SLYKE. I withdraw it. I believe those are all the recommendations.

Mr. WILEY. I move to strike out the reference on page 183 to a method of analysis as I do not think the reference is necessary.

Adopted.

Mr. DE ROODE. We ought to do away with the condenser. I move that all about the condenser be stricken out.

Carried.

Mr. SCOVELL. I move that a committee of three be appointed to report to this association this afternoon on nomination of officers, and I insist that I shall not be on that committee.

The president appointed as such committee Messrs. Winton, Hutchinson, and Huston.

Mr. WOODS. It is a matter that I am not specially interested in, yet it seems to me that since we yesterday hastily adopted the Gunning method the reporter for the next year should be instructed to study into a method whereby the Gunning method can be adopted for the use of fertilizers containing nitrates.

Adopted.

Mr. DE ROODE. When we were on the subject of phosphoric acid I showed some figures comparing the results obtained by the Gunning method adapted to phosphoric acid, and I would like to have that referred to the phosphoric-acid reporter next year, or to the nitrogen reporter. There seems to be a misunderstanding about that, as each of the reporters says the other has it.

The PRESIDENT. Which do you prefer?

Mr. DE ROODE. I would say the phosphoric-acid reporter.

The association, at 12:30 p. m., took a recess to 2 p. m. Friday, August 26, 1892.

AFTERNOON SESSION, FRIDAY.

The association was called to order by the president at 2 p. m.

Mr. WILEY. I have a letter from Prof. Jordan, of Maine, announcing his inability to attend on account of illness. His report on foods low in carbohydrates I handed to Mr. Anderson at Mr. Jordan's request this forenoon to look over and he has not come in yet. I also have a letter from Dr. Stubbs, of Louisiana, regretting his inability to attend on account of illness.

Mr. Harrington was then called on to read his report on soils and ash analysis.

REPORT ON SOILS AND ASH.

By H. H. HARRINGTON.

One sample of soil and one sample of wood ashes were sent out to such of the chemists as would agree to undertake the work. Also a "soil solution," prepared from carefully purified chemicals and supposed to represent the acid extract of the soil. Of the number of chemists who agreed to undertake this work, seventeen in all, but two reported on all the work, and two others on a part of the work.

The reporter felt, therefore, some hesitancy in recommending changes on adopted methods, even when the necessity of such changes was convincing to his own mind, since the methods had received attention from only three or four different laboratories.

In sending out my circular letter, directions were given to carefully follow out the official method, when practicable, and in case of alternate methods, state which was used; or in deviating from the official method, accurately describe the plan followed.

In my own laboratory we followed the official method, as near as possible, except in case of manganese, and we were entirely unable to make an estimation of this in the manner prescribed.

Prof. Peter, of Kentucky, has this to say in regard to his work:

"In the soil and soil solution I have followed the method in Bull. 31, U. S. Department of Agriculture, Chemical Division, though I took 14 grams of soil for nitrogen, instead of 20 grams, and used only 1 gram sodium thiosulphate, the nitrogen in soils being so small even the above quantities of reagents are obviously much larger than is ever necessary. In the ash I have departed somewhat from the prescribed method. The method followed was to digest 5 grams in HCl as directed, evaporating to dryness to separate silica, and making up to 250 cc.

(1) 100 cc. of this solution were neutralized with NH_4OH, and treated by the molybdate method for phosphoric acid.

SOIL—BRAZOS RIVER SILT.

	Water.	Organic and volatile.	Iron.	Alumina.	Manganese.	Lime.	Magnesia.	Potash.	Soda.	P_2O_5.	SO_3.	CO_2.	Total N.	Si O_2.	Soluble. SiO_2.
Peter (Kentucky)	3.68	2.50	3.21	7.68	.....	5.62	1.86	1.10	0.23	0.14	.....	3.99	0.0570	68.450	.....
De Roode (West Virginia)	3.57	2.58	3.35	7.36	.....	5.59	1.96	1.01	0.22	0.11	0.07	.....	.0560	68.620	.....
Tilson (Texas)	3.24	2.58	3.63	5.58	0.22	5.61	1.84	0.81	0.59	0.39	0.30	4.11	.....	70.940	.....
Adriance (Texas)	3.26	2.68	3.61	5.58	0.22	5.66	1.85	0.88	0.22	0.34	0.29	3.99	.....	70.920	.....
Collingwood (Arizona)	.....	.....	3.53	9.20	.....	5.42	1.93	1.19	1.35	0.21	0.36	.....	.....	68.596	14.79
Eaton (Arizona)	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.0535	67.930	.....

ASH.

	Water.	Organic and volatile.	Iron.	Alumina.	Manganese.	Lime.	Magnesia.	Potash.	Soda.	Phosphoric acid.	Sulphuric acid.	Carbon dioxid.	Silica.
Peter	.....	Trace.	0.87	.....	0.47	47.78	2.36	4.29	0.59	0.64	2.37	31.24	7.75
De Roode	.....	Trace.	0.93	.....	0.47	47.46	2.51	4.27	0.50	0.63	2.44	.....	7.67
Tilson	.....	.29	1.06	.....	1.01	47.22	2.70	4.34	0.52	1.20	1.96	31.36	7.77
Adriance	.....	1.14	1.02	.....	1.10	47.32	2.62	4.62	1.44	1.13	1.88	31.32	7.72

SOIL SOLUTION.

Peter	.....	.....	1.86	1.56	.....	2.47	1.12	0.28	0.22	0.56	0.29	.....	.....
De Roode	.....	.....	1.97	1.19	.....	2.55	1.16	0.28	0.25	0.59	0.27	.....	.....
Tilson	.....	.....	2.05	1.34	0.24	2.53	1.33	0.21	0.11	0.53	0.17	.....	.....
Adriance	.....	.....	1.98	1.27	0.26	2.54	1.33	0.28	0.11	0.53	0.14	.....	.....
Patrick (Iowa)	.....	.....	1.87	1.66	.....	2.45	1.35	0.27	0.09	0.47	0.20	.....	.....

THEORETICAL SOLUTION.

.....	.....	.....	2.00	1.42	0.22	2.50	1.327	0.252	0.113	0.50	1.56	.....	.....

Mr. Eaton also made determination of ammonia in the soil by Schlösing's method (Grandeau) and "total available," extracting as in Schlösing method for NH_3, reducing by copper-zinc, and distilling.

	Silica.	Solution.
Hilgard's method	68.02	15.56
Hilgard's method	67.25	.....

(2) 50 cc. were treated for SO_3 by $BaCl_2$, the filtrate was evaporated to dryness, the residue dissolved in water and treated with barium hydrate for alkalis.

(3) 50 cc. were neutralized with Na_2CO_3, a few drops of Fe_2Cl_6 added, and precipitated by boiling with the addition of sodium acetate. In the filtrate, the manganese was precipitated with bromin, the lime and magnesia as usual. The lime was weighed as CaO, as was done also in the soil and soil solution.

Two grams of the ash were treated in a Sheppard CO_2 apparatus for CO_2, using dilute HCl. The solution was evaporated with H_2SO_4, reduced, and the iron treated with permanganate. Prof. Collingwood says: "In the method for iron I found it impossible to dissolve the fused mass in conc. H_2SO_4—tedious and unsatisfactory in HCl."

We made no estimate of nitrogen, but the results reported from Kentucky and Iowa agree very closely.

The following is the report of the work done:

This is the first time results have been reported on soil and ashes by the association. In many cases the difference is very wide, but take the work in its entirety, and remember the small number of reports received, together with the fact that this was really the first extended trial of the method adopted last year, and the figures here reported make a gratifying showing.

I sent out three samples and tried not to slight anybody, but found difficulty in getting the work done. This is the first report, I believe, on soils, last year being the first time that a method had been adopted by the association. The work is of course tedious and laborious so far as the details are concerned.

The first sample is soil from the Brazos River—silt. We will notice the soil first as to water. These determinations are generally satisfactory.

In alumina the results were unsatisfactory, decidedly.

Manganese was unsatisfactory and I have a recommendation in reference to it.

Lime is generally satisfactory. Magnesia is also.

Potash is somewhat unsatisfactory, but still very good for the first reports on the work.

Soda was satisfactory with the exception of that from Mr. Collingwood, of Arizona.

Now, then, you will turn to the second sample:

They are fairly satisfactory except in the case of manganese, which I will refer to in the same way that I will for determination of soils, the official method for manganese being unsatisfactory, to my mind.

Lime is fairly close.

The phosphoric acid is unsatisfactory.

The sulphuric acid is fairly good.

The carbon dioxid is fairly so. There have not been as many reports as I should like, but you see under the head of Soil Solution they are satisfactory so far as the work was done. Taking iron, the first case, the results are all that could be expected on so many determinations where iron, alumina, manganese, lime, potash, soda, etc., are all to be determined.

Now we come to the recommendations:

RECOMMENDATIONS OF THE REPORTER.

The first recommendation is, commencing after the semicolon, about half way down the second paragraph, page 233, Bull. 31, commencing with the words "Pass 8 to 10 pounds of the soil through a sieve having circular perforations one-twentieth of an inch in diameter," right after that I would add the following: "Noting the percentage of the amount of soil rejected." If the amount equals 20 per cent or over this should be pulverized to pass through a sieve of one-tenth inch circular holes, and in this state submitted to analysis. That is simply to provide for gravelly soil, soils where the amount of coarse material is very great. This extra analysis would not generally be necessary, but might become so sometimes.

On page 236, "Acid digestion of the soil," down to the words "Temperature of boiling water," I have somewhat radical recommendations to make. That is to "place in boiling water and digest for two hours at this temperature." I believe that is a sufficient length of time to take out all the iron in the soil and leave a fine white residue.

I do not claim that it gets out all the soluble material, nor do the thirty-six hours, but the thirty-six hours will get out five-tenths more of soluble material, and the only way to get out all the soluble material would be to fuse the soil and take that. Dr. Hilgard, as I have just read you, and as is well known, digests for five days and for ten or twelve hours a day; but, as just read you and as you see on the tables, that gives very little difference from the thirty-six hours' digestion. I have tried a number of soils with this two hours' digestion, and the difference is only about one-half of 1 per cent—that is to say, the two hours' digestion gets about one-half of 1 per cent more of insoluble material, but the residue is white, and the iron of perhaps half a dozen soils I tried was fully extracted.

In that same paragraph, commencing at the words "pour the contents of the vial into a small beaker," and down to the words "make filtrate up to 500 cc."—now, instead of that, I would recommend that you wash with hot water, allowing the washings to run into the filter, etc. As soon as digestion has taken place, wash it, dilute it if you think it is necessary, or filter it immediately and wash with hot water. All that residue will represent the insoluble matter in the soil; evaporate the filtrate, after adding few drops of HNO_3, to dryness, etc., and filter to get the soluble silica; that may not get all the soluble silica—that is to say, there may be some soluble silica remaining in the total silica that can be separated if desired. I will come to that in a moment.

According to pages 236 and 237, sections 13 and 14, I have had great difficulty in getting dry precipitate to dissolve in sulphuric or hydrochloric acid; therefore I would recommend taking a separate portion of solution and precipitate 100 cc., and that it be washed with hot water. Dissolve while still wet, and you have no difficulty in getting it in solution in that state.

I have found the greatest difficulty in determining manganese by the official method. The temperature specified is 60 for twenty-four hours; with that temperature I have been unable to get manganese to precipitate, and you will notice from the figures handed around that manganese is unsatisfactory all the way through. The results vary more widely on that than on any other determination; therefore I would recommend the solution to be alkaline and kept near the boiling temperature, with fresh addition of bromin water until the manganese precipitates. It will require a little perseverance to do that. It should also be filtered while hot. I continue adding bromin water for perhaps an hour, keeping it at boiling heat during that time. It need not be boiling vigorously, but near a boiling temperature. I filter it while hot, and generally succeed in getting satisfactory results.

In case of lime you will notice one principal method and two alternate methods. I should recommend precipitating as oxalate. Filter, dry and ignite to constant weight. Weigh as CaO.

The phosphoric acid has been included under the head of "Iron."

In the case of nitrogen I have no recommendations to make, not having tried the adopted method sufficiently. Only total nitrogens were made, but the results agree very closely. You will find the method of total nitrogens on page 240.

It might be well to call the attention of the next reporter to the method prescribed on page 235. I do not attach very much importance to that, as it does not seem to me to have any very great importance; but, of course, there must be a method given. It is desirable sometimes to determine the amount of nitrogen in soil, but except for a total amount the determination can not be of any consequence.

In the method for ash determination, on page 241, at bottom of the page, last paragraph, where it says "with substances rich in silica and alkalis it is better to first char the substances," I would recommend putting in there "rich in silica, alkalis, and phosphates, including foods and materials of that kind," such as are designated on page 242.

On page 242 there are two methods for that. I would recommend the alternative method.

In carbonic acid, under the fifth section, on page 243, I would take the original substance instead of heating first, and weigh out 2 grams, or whatever may be considered necessary, leaving that to the opinion of the chemist to determine by the amount of carbonic acid expected to be present. The alkalimeter described gives fairly good results. The amounts reported on the paper are concordant. I like the apparatus described by the Department of Agriculture in Bulletin No. 13, Part IV.

In case of chlorin I recommended to precipitate as the silver chlorid, and filter through a Gooch crucible, and dry at 110°. That gives very satisfactory results in air bath and then weighing.

On page 244 I would recommend using some other solution and, if it is necessary to increase the quantity of ash, so as to increase the quantity of the solution digested, taking only five grams, or you can take, say, ten grams and double the quantity of acid.

I believe this is all I have to say.

The PRESIDENT. I am sure that I but voice the sentiments of every member of the association in giving expression to our gratification at having the honorable Secretary present with us. We will be very glad to see him here during any of the future meetings of our association. We will be pleased to have a few words from him.

The president introduced Hon. Edwin Willits, Assistant Secretary of Agriculture, who spoke as follows:

ADDRESS OF HON. EDWIN WILLITS.

Mr. PRESIDENT AND GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: I am glad to look in upon your deliberations again. The Department of Agriculture appreciates very highly the work which you have performed, and it looks with interest upon the results of every meeting which you hold. In truth, we consider you, gentlemen, in the labors that you perform, in the investigations you make, as contributing very largely toward the work the Department itself is performing. We find that you are colaborers with us, and very efficient ones. I can not let this moment pass without looking in upon you in behalf of the Department to encourage you to proceed in your labors, to continue your investigations, and perfect more and more your work. Your work is important. It is, morever,

important that you, by this coming together of so many workers, should work out as far as possible a common standard.

Chemistry is not, in all of its relations at least, an exact science, and yet by constant communication with each other and comparison of results you may make it more and more exact. Then, again, you have comparisons of different conditions. Chemistry as applied in one locality seems to be modified and influenced more or less by conditions that exist in other localities, and various influences have to be taken into account. Then, again, your meetings here have in large measure brought about and will in still greater degree bring about an adjustment of personalities, of individualities, because it is in your work as in almost all other lines that the individual some way or other impresses himself upon results, and that with the same formula, the same material, the same supposed conditions by different individuals different results will be brought about. The more you meet, the more you confer together, the more you examine your results, the more and more you will come to a true knowledge of your limitations, the limitations of chemistry, and you will form a more abiding and true conception of those limitations and where those limitations exist. This is especially true of agricultural chemistry. It has come to your knowledge and largely to your high appreciation that chemistry is not the sole ultimate authority to the extent it was once thought to be; that there are other factors that come into operation that modify results which were not known or appreciated fully in generations past. These factors, influencing chemical operations, may be made a study, and you certainly can take them into consideration.

They will account in a large measure for differences of results of the same formula or the same constituents. Again, it is obvious that nature is working, competing, coöperating with you in your results through organic life, through bacteria, so that now the idea has grown up that your results are not conclusively obtained until you have called to your aid the microscope. Again, every animal, every plant, is itself a laboratory, but the digestive laboratory of a plant or an animal is not, of course, the exact counterpart of a chemical laboratory. This study of life, of organic life, upon these chemical agencies, upon these constituents, has its effects, and all have to be taken into account. Your researches, so far from being narrowed by means of these other agencies, are widened so that they take in other sciences and other influences that were not thought of a generation ago. But, after all, while it is true that physics and biology are being urged ahead in the investigations of the world, the scientific world, nevertheless chemistry, as in mathematics and the study of all the forces of nature, has made great strides, and I have words of encouragement for you to go ahead in your work. Taking all these considerations into account, your results will be more complete in the future. You can prophesy what shall come to pass more truly than you could in the old set formula

under the idea that chemistry solved everything. Again, I say, I am happy to meet with you on this occasion. Notwithstanding the great strides chemical investigation is making all along the line, in the results as they come to us from year to year, there is consolation that, though you may study from year to year and generation to generation, there is constantly an opening and widening field that will amply compensate you for all your labor and will inspire you to higher efforts.

Hoping that you will have, as in the past, a successful and instructive deliberation here, I meet you here and greet you in behalf of the Department of Agriculture, and wish you long life and prosperity in the future. [Applause.]

The PRESIDENT. We will now proceed with the subject that we have under consideration. However, it is but five minutes to 3 o'clock, when the special order is to be considered.

Mr. BABCOCK. I move that we now proceed with the miscellaneous business which was made the special order for 3 o'clock.

Adopted.

The president vacated the chair, having called Mr. Babcock to preside over the association.

The ACTING PRESIDENT. We will now proceed with the special order. We had a committee appointed on the selection of officers for the coming year, and the report of that committee will be in order.

ELECTION OF OFFICERS.

Mr. WINTON. Your committee on the nomination of officers reports as follows:

For president, Mr. S. M. Babcock.

For vice-president, Mr. E. B. Voorhees.

For secretary, Mr. H. W. Wiley.

For executive committee, Messrs. Kilgore and Harrington.

The PRESIDENT. It is moved that the secretary cast the ballot for the gentlemen named.

Carried.

Mr. WILEY. I have cast the ballot as instructed.

The PRESIDENT. The ballot has been cast by the secretary, and the officers mentioned in the report have been elected for the ensuing year.

MISCELLANEOUS BUSINESS.

Mr. SCOVELL. I offer the following resolution:

Resolved, That this association appoint a committee of three to act in conjunction with committees appointed by the American Chemical Society, the Chemical Section of the Association for the Advancement of Science, and the World's Columbian Auxiliary for the purpose of securing an international congress of chemists during the summer of 1893 at Chicago.

It was adopted.

Mr. SCOVELL. I would suggest that the chairman be president of that committee.

Seconded.

Mr. PAYNE. I would like to offer a resolution, if it is the proper time to do so, that our secretary be instructed to prepare a list of the names and addresses of all the official chemists of the United States. When the reporter wants to send out samples, and wishes to send them to all official chemists, it is very difficult to find out who they all are. It ought to be headed "List of official chemists of the United States; who they are and where they are located."

Carried.

Mr. WILEY. I feel that it will be somewhat difficult to prepare such a list unless we know more specifically what is meant by "official chemists." I suppose it is meant to include chemists connected with experiment stations, agricultural colleges, boards of health, and other municipal and State bodies having control of the products mentioned in our constitution. I have such a list, a mailing list of persons coming under this head, amounting to about 250 names, and I suppose that list will include all that are meant to be included in the resolution. There will be no difficulty in publishing that list in the proceedings, if it is so desired.

The PRESIDENT. I think that is probably what was intended by the resolution. There are a great many chemists, who hold semiofficial positions in the country, whom it would be impossible to include without a great deal of trouble, and I do not think that could have been intended by this resolution.

Mr. PAYNE. My idea was to have a list of those chemists who join us in our work, so that we could compare our work, so that we may know the men that we are in contact with. When I got up my record as a reporter I was desired to state the official position of each one taking part in the work, but I could not ascertain it in all cases.

The PRESIDENT. Is there any other business?

Mr. LORD. There is a resolution which I think ought to be offered in regard to the directions of the official methods. There is a number of minor points that are made difficult each year by reason of the indefiniteness of the recommendations of the reporter or from errors which are largely typographical. I understand, from conversation with Dr. Wiley, that under our present rulings he considers that he has not the power to change a single word nor even a comma in the official methods. For that reason I move that, in printing the official method and in publishing the proceedings of the meeting, the secretary be authorized to make such minor changes in the wording as shall seem desirable, provided no alteration in the method of work or the description of method is involved. In other words, that he be allowed to edit the matter.

Adopted.

Mr. SCOVELL. I see under the head of official methods a note by the secretary, in which he says that he has hard work to get the reports from the different reporters so that they can be promptly printed, and he requests that the constitution be changed so that he may be able to get the correct report. I would suggest that the reporters be requested to hand to the secretary the correct method as soon as possible each year, and that they also include in those reports a résumé of the publications in this or foreign countries during the year on subjects connected with their several topics of investigation.

Mr. LORD. In regard to the abstracts, I would say that, as the reporter on phosphoric acid, I have prepared no abstracts for the last year. I understand some other reporters have omitted the work. The preparation of abstracts by the reporters is sometimes a matter of great difficulty. Now, in my case I, of course, would have given abstracts of a number of papers that came under my notice, but I felt that with my facilities for access to journals such abstracts would not be as complete as they should be to put into such a publication; and it strikes me that perhaps it ought to be well to have that work done separately or at some place where the best possible opportunity for such comparative work could be done. It should be very complete, and a partial one would simply be misleading. I throw that out as a suggestion.

Mr. VAN SLYKE. My own experience is much as Mr. Lord's. In my case it would have been impossible, without going to some large library, which is not readily accessible, to prepare work that would be at all satisfactory; and while I think it is desirable that it should be done every year, still we will run across cases probably every year where it is practically impossible for the reporter to do so. Of course, in times past, where it has not been practicable for the reporter, he has arranged with somebody else who did have access to a large library to do the work, but to require every reporter to make a bibliography every year would not accomplish the aim we are working for.

Mr. PAYNE. I will have to join the other three gentlemen in their remarks. I gave out all the information I came in contact with, but there may be a great deal that I did not see.

Mr. HARRINGTON. As reporter on soils I will say that there is a great deal of material in the shape of analytical work that might be put under that head and be published, and, at the same time, I could not undertake to publish a full review of everything upon that subject, because I have not facilities for so doing. Of course I could make arrangements to have it done at considerable trouble or expense, but it seems to me that would be a matter that ought to be left to the individual opinion of the reporter. These abstracts are now published by the Office of Experiment Stations; at least the abstracts of foreign literature are to some extent, and we get a good deal of what is being done from their reports. I think that the first part of the resolution is very desirable—that the reporters should furnish the secretary with

a report of their work ready for the printer; but I should not be in favor of compelling each reporter to give the abstracts, for there might be and certainly would be a great difference of opinion as to what that literature would include, how much should be included, and where the line is to be drawn on the literature.

Mr. FREAR. The reporter does about all his official work will permit, and still it is true that each of us who renders services to three or four or five reporters finds his hands pretty well filled up. I suggest the appointment of a committee on abstracts. I believe that we might get a more satisfactory division of the labor in that way. That committee must be selected with reference to the subjects. I feel very much inclined to move that, in place of assigning this work to reporters, it be hereafter assigned to a committee on abstracts.

Mr. SCOVELL. I simply put a motion requesting the reporter to furnish abstracts. The reporter generally is a man who looks up what methods others have done in his line of work. When I was a reporter one time I got somebody else to make the abstracts for me, and so did Dr. Jenkins, when we did not have the literature ourselves.

Mr. PRESIDENT. I think myself, if there is any danger of this last portion of the resolution defeating the whole, that it should be divided.

Mr. SCOVELL. I call for a division.

Mr. PRESIDENT. You have heard the motion: That the reporters be requested to prepare their reports in form for publication, together with any recommendations and amendments which were made, and also that the reporters be requested to make as complete a bibliography as possible. I think that covers the subject.

Mr. FREAR. I would like to call attention to my motion, and that is that this subject of bibliography be referred to a committee on abstracts, to consist of not less than five, to prepare the abstracts on agricultural chemical analyses.

Seconded.

The PRESIDENT. The amendment will have to come first.

Adopted.

Mr. WOODS. Is it possible to have the motion stated as it now stands?

The PRESIDENT. The motion as it stands, as I understand it, would be that, first, we have a committee appointed, that the reporters prepare their reports for publication, including the emendation and recommendations; and this has been amended with the report that a committee be appointed for abstracts, which is attached to the original motion as it stands.

Mr. SCOVELL. I would like to vote for one, but would not like to vote on the other—there is no relation between them; therefore it should be voted down.

The president submitted the motion and decided that the whole resolution was lost.

Mr. SCOVELL. I now move that the reporters be requested to hand to the secretary as soon as possible their report, together with the method as adopted for use during the ensuing year.

Adopted.

Mr. FREAR. I move the adoption of the second part of that motion—for a committee to make the abstract on the subject relating to chemical analyses.

Adopted.

Mr. WILEY. During the hour for miscellaneous business, I would like to make a suggestion, or, at least, make a statement, in regard to our meeting next year. Several have spoken to me about the desirability of holding our next meeting of the association in connection with the World's Columbian Auxiliary Association, or in connection with the International Congress of Chemists, which will certainly be called to meet in Chicago next summer. I do not think there would be any objection on the part of the United States Department of Agriculture next year to holding the meeting in Chicago. If it is the desire of the association, I have no doubt the executive committee, which has this matter in charge under the constitution, would appoint such a meeting. I think it would be well, if we could have an expression from this association by which the executive committee could be guided in this matter, because it is one of considerable importance. So far as I am personally concerned I have no preference in the matter. I shall certainly attend both meetings, if possible, whether held distinct from each other or conjointly.

Mr. PAYNE. Do you know the date of the meeting referred to?

Mr. WILEY. It is about this time of the year.

Mr. HUSTON. I am in favor of holding our meetings here, as I think our work would be of a better character than if held in Chicago at the time of the World's Fair.

Mr. WILEY. On consultation, I find that the association has to determine the place of meeting and that the executive committee has nothing to do with it, only the time of meeting; therefore, it is the duty of this body to fix the place for the next meeting or confer that authority by direct vote upon some body like the executive committee.

Mr. FREAR. In order to bring this matter before the association I offer the following:

Resolved, That the next meeting of the association be held in Chicago.

Personally, I very much prefer that it should be held there.

Mr. VAN SLYKE. I support the motion, in order to get it before the meeting.

Mr. WOODS. I move to amend by substituting the word "Washington," in place of the word "Chicago."

Seconded.

Mr. KEDZIE. I move as a substitute for the whole thing that the time and place of the next session be left in the hands of the executive committee.

Adopted.

Mr. WINTON. I move that the reporters be requested to send out to the members of the association their samples and circulars and other matters by the 1st of December.

Mr. WILEY. Allow me to suggest an amendment; say December 1, or as soon as possible after the receipt of the published methods of analysis adopted at the meeting.

Adopted.

Mr. HUSTON. I offer the following:

Resolved, That this association convey, through its secretary, the thanks of the association to the authorities in charge of the National Museum for the use of this room for our meetings, and to the officers of the Department of Agriculture, especially to Assistant Secretary Willits, for their efforts which have contributed so much to the success of this meeting.

Adopted.

On motion the regular order was resumed.

The PRESIDENT. We will consider the recommendations of the reporter on soils and ash. Are there any remarks?

Mr. ROSS. I notice that under the head of analysis of soils used for solution that only 5 grams are specified, and out of that only 2 grams are taken for phosphoric-acid examination. It seems to me this is a very small margin.

Mr. ROBINSON. In estimating phosphoric acid in soils it doesn't come into the estimate from 5 grams until you reach the third or fourth place of decimals. The man who decides on phosphoric acid in so remote amount must do it more skillfully than I can. In point of fact in some solutions of soils the mineral material in solution does not exceed 2 per cent, and about four-fifths of this are iron; and you have to make an estimate of the other materials, of course, according to this proportion. In point of fact 5 grams is utterly insufficient. Ten times 5 grams are nearer the amount that is absolutely required for accurate work. I have been using for the determination 75 grams of soil. I defy any man to make an accurate estimate of potash and soda with 5 grams of soil in some of our Florida soils.

Mr. HARRINGTON. I would only say in regard to that that I have never been in favor of specifying particular weights which a solution shall carry. That seems to me a matter that should be left to the chemist and the character of the soil. You can not prescribe all the minutiæ for any soil. All we can do is to lay down rules for average soils. The recommendations I have made are to determine the potash and soda from the sulphuric acid, the sulphuric acid being determined from the direct solutions. I have no objection to increasing it to a hundred grams, so that you have to decide upon one or the other anyway, and that was all I tried to arrive at.

Mr. FREAR. It seems to me that the method of the analysis should be looked to carefully, but I did not notice any recommendations of the reporter referring to the sampling. I move that the directions given by Dr. Hilgard for sampling the soil be substituted for the directions recorded in the official method.

Seconded.

Mr. HARRINGTON. I offer, as a substitute, that on the third line from the second paragraph it should read: "Down to the depth of 6 or 9 inches, or until the character of the soil indicates a change from the top soil to the subsoil." I noticed that error myself, and I supposed that would be taken for granted.

Mr. FREAR. I do not care to go down further than 9 inches.

The PRESIDENT. Why not leave out the 6 or 9 inches?

Mr. FREAR. Make it "or unless the surface soil be more shallow."

Mr. HARRINGTON. I have no objection to that amendment.

The PRESIDENT. It is moved that in the second paragraph of the method for sampling soils it be amended to read, in the third line, "taking it down to a depth of 6 to 9 inches unless the surface soil appears more shallow." I think that is the way it stands now.

Adopted.

Mr. ROBINSON. I have no other suggestion to make in regard to that paragraph, but there is one other factor in drying which seems to deserve attention, and that is the humidity of the atmosphere at the time you are drying.

The PRESIDENT. Was there any motion in regard to that? If not, we will proceed to the next.

Mr. ROBINSON. I only made that as a suggestion.

The PRESIDENT. If not, we will proceed to the next suggestion. We will take them up by paragraphs, in the order in which they occur.

Mr. HARRINGTON. There is one recommendation that has not been acted upon. I am not particular about it. It is in the first paragraph: "To eliminate, etc." down to the words "averages of the soil." It has been the general method adopted, but it does not seem to me to be quite the thing to do to get the sample for analysis.

The PRESIDENT. I do not think a motion is necessary in regard to these recommendations, and unless there are objections, and as they are stated I will call for a vote; it will save time.

Mr. HARRINGTON. Let that stand as it is, then. The next is the middle of that paragraph: "Pass 8 to 10 pounds of the soil through a sieve," I make an amendment in the percentage of the amount of such soil rejected. The further recommendation is if that amount exceeds 20 per cent or more that it pass through a 2.5 millimeter sieve, or whatever size is desirable, and then that amount be submitted to analysis. Heretofore the percentage of the amount has been noted and it has not been analyzed.

Mr. WILEY. I second the suggestion which has been made in regard

to introducing the metric system of measurements. In addition I will say, while on this point, that I have received a letter from Dr. Hilgard, and he is strongly of the opinion that the half-millimeter sieve is the proper one.

The PRESIDENT. Now comes the suggestion in regard to the half-millimeter sieve.

Mr. WILEY. I suggest that it be referred to the reporter with a request for investigation during the coming year.

Adopted.

The PRESIDENT. Now we come to the next recommendation of Mr. Harrington, that when the amount exceeds a certain per cent that it be analyzed by itself. Mr. Harrington will state that again.

Mr. HARRINGTON. "Noting the percentage of the amount of the soil rejected." If the amount equals 20 per cent or over this should be pulverized to pass through a sieve of 2.5 millimeters circular mesh and in this state submitted to analysis.

Adopted.

Mr. HARRINGTON. The next recommendation is, page 236, under the head of "Acid digestion of the soil," to digest two hours instead of thirty-six.

Mr. WILEY. That is such a radical departure that I think the best we can do with the recommendation is to commend it to the reporter next year for further investigation.

Carried.

Mr. HARRINGTON. The next is on page 236, under section 11, on the line below the words "thirty-six hours", I recommend, "filter and evaporate and wash with hot water and allow the washings to run into the filter, burn the residue, and calculate the insoluble silica."

Adopted.

Mr. HARRINGTON. The next is at the top of page 237. For that I recommend "to precipitate a fresh portion and dissolve while in a wet state," etc.

Adopted.

Mr. HARRINGTON. The next is in regard to manganese, page 237, middle of the paragraph. I recommend a fresh solution of alkali, added until the manganese precipitates, filter while hot, adding bromin water for half an hour, keeping it at a boiling heat during that time.

Adopted.

Mr. HARRINGTON. Under the head of determining total nitrogen by the Kjeldahl method, I would recommend the use of the methods which have already been adopted by the association.

Adopted.

Mr. HARRINGTON. The next is on page 242, under "analysis of wood ashes," in section 4, "phosphoric acid," I would recommend the official method.

Adopted.

Mr. HARRINGTON. In the case of chlorin (page 243) I would recommend the precipitation as silver chlorid and filtering and drying and weighing in the Gooch crucible.

Adopted.

Mr. HARRINGTON. For lime there are three methods. I would recommend the same method for determination of lime as in soils.

Adopted.

Mr. HARRINGTON. From an oversight, carbonic acid was not included in the soils last year, and I recommend that the same methods be used as under the head of ash.

Adopted.

Mr. HARRINGTON. That is all. I move that the report be adopted as a whole.

Mr. DAVIDSON. Did you leave out the alternative method for determining the chlorin in ash analysis?

Mr. HARRINGTON. Yes.

Mr. DAVIDSON. I move to have it put in.

Mr. PETERS. I do not think it would be advisable to adopt this report as an official method. Mr. Harrington has done as well as he could, but the methods are yet under trial. We should give the reporter next year further time for consideration.

Mr. WILEY. Then your motion is to adopt the report as amended as a provisional method of analysis for the coming year?

Mr. PETERS. Yes.

Adopted.

Mr. WILEY. I desire to have the association listen for a short time to Mr. Milton Whitney, in explanation of some apparatus that he has devised for taking soil samples. Mr. Whitney has come from Baltimore especially at my suggestion to present these matters to the association and did not get here in time to be present when we considered soil sampling, and I suggest, therefore, that the president ask him to now present this matter to the association. You will find the matter exceedingly interesting.

Adopted.

Mr. WHITNEY. I am very sorry I did not get here in time to hear the discussion of the methods of soil analysis, but there was a meeting of the trustees of our station in Baltimore and I did not succeed in getting over in time to hear the discussion which I was very anxious to hear. I have brought with me the auger and other apparatus I use in taking samples of soil. The old method of collecting samples of soil with a spade is exceedingly laborious. I have found it so myself in tramping over several States and in Maryland last year. I went on a trip through southern Maryland with a long-handled spade and walked 6 and 8 miles with a knapsack loaded as it is now weighing from 15 to 25 pounds, and in addition I had to carry my samples myself. I found I could take but a very few samples. I use this common

wood auger that I had fixed up for this purpose. It was an ordinary 2½-inch wood auger that I had lengthened a little and fitted with this short piece of gas pipe for a handle, which can easily be slipped out and put in the knapsack when not needed. This is easier to carry on a walking or driving trip than a spade, and with it I can readily sample to a depth of 18 inches. In taking a sample of the soil proper I usually go to the change of color where there is a difference between the soil and subsoil, which is usually to a depth of about 6 or 9 inches. The auger takes up a very nice sample, except that in a dry, sandy soil there is a little trouble in getting it up, but I can always clean out the hole and the subsoil is always so tenacious that it clings to the auger, and I can easily get it up and put it in my bag. After taking a sample of soil I clean out the hole and then I go down in the same place for the subsoil and take the sample to the depth of 18 inches from the surface.

I have two sets of sacks that I use for the samples. For many of my samples I use a sack of the size of the one I have in my hand, 6 by 8½ inches. This auger once filled with a soil is not quite enough to fill that bag, but I get plenty to fill it in the deeper subsoil sample. When I want a larger sample I have a larger sized bag (8½ by 15½ inches) that holds several times as much as this one. These bags are made of heavy cloth, and when the moist soil is put in them it makes the sack almost perfectly tight until I get to the laboratory. I have tried to use glass jars for sampling, but it is almost impossible to carry them around, and even in the laboratory and storeroom it is impossible to keep so many large samples in jars. With these sacks and the auger I can take fifty samples a day on a trip. Then I have a label here that I attach to the sacks. I number all the sacks on the outside and put a number on the label to correspond to the number on the sack.

The auger could be of any size, but I think 2½ inches is the best size. I think this is as large as can well be used. It can be of any size, however. Six inches in depth of soil or subsoil is about as much as one wants to pull out, especially in heavy clay. I always find when I get to taking subsoil samples that I have to pull the auger up and empty it after going 3 or 4 inches.

I never aim to take many samples in the same field. I sample localities more in the work I have been doing. I have a geological map that I use, and I am very sorry I forgot to bring it over. I know the localities where I want to go and I know the area of the soil, and especially the kind of soil, that is, for instance, whether it is limestone or not. I know from former experience or from a preliminary survey the general character of the soil and I select from as widely different localities as possible. I do not aim to take in any particular locality. I am always careful to reject any sample or any spot where there are evidences of local discrepancies, washings, or anything of that kind, where the soil can not be considered representative of the particular soil formation, leaving the consideration of these local questions until

the general problems have been worked out. Thus I will drive, perhaps, a good many miles, but still keep in that same formation, and the general character of my soils will be very uniform. I may drive 10 or 15 miles, or take a car and go to any other part of the State, and I know that in the same formation the samples I take will be quite uniform with those from the first locality. They run very uniform in our eastern and southeastern States, so that if I am sampling from our Cumberland Valley limestone, for example, I would get localities, not from, the seveeral places on the same farm, but from as widely different localities possible; perhaps twenty-five or fifty localities in all that would be examined separately.

In the next clauses of this method of soil analysis, on the "relation of soil to water," "hygroscopic moisture," "capacity to hold water," I would like to show a little apparatus I have here and always take with me in my fieldwork. The relation of the soil to water will depend largely, as I have pointed out in various papers, on the amount of empty space there is in the soil, that is, the amount of space that is filled with water if the soil is saturated; and also on the number of grains and the amount of subdivision of that space, and the way the grains are arranged. To determine the amount of empty space I use the form of sampling tube that I have in my hand. It is a brass cylinder with a piece of clock spring soldered in one end and turned off in a lathe to give a good cutting edge of steel. I find where I use a simple tube that the friction of the soil on the inside of the tube is so great that the level of the soil on the inside of the tube is often an inch or half an inch lower than the level of the soil outside, so that I had this watch spring put in to avoid this friction. I determine the area inclosed by this clock spring very accurately, and then I make a 6-inch mark on the side of my cylinder, which is 9 inches long. The cylinder might be just 6 inches long but I have it 9 inches long so as to have a little projection to take hold of. I drive it into the ground with a hammer to the depth of 6 inches, using a heavy steel cap to receive the blows, and then I dig it up with this small trowel, being careful to pass this broad-bladed knife under the cylinder when I lift it out to keep the soil from falling out. I put the soil into one of these small sacks and take it to the laboratory and spread it out on paper and air dry it, and when it is air dried I weigh it. The volume of the soil removed is as large as the area inclosed by this steel spring and 6 inches long, and is just 302 cubic centimeters. Now, if I know the specific gravity of the soil it is a very easy matter to find out the amount of space: to divide the weight of the soil by the specific gravity which will give me the volume of the soil pretty closely, and after thus finding the real volume of the soil grains, I subtract this from the total volume of the sample (302 cc.) and find the amount or the per cent by volume of the empty space in the soil. Of course it would be well to determine the specific gravity of the soil in every case, but I do not do it. I use the factor

2.65, and this is close enough for the present stage of soil investigations. I take a sample of this kind to determine the per cent by volume of empty space to give an idea of the compactness of the soil whenever I take a sample of soil, if possible. You will find, if you try to make any calculations of the relations of soil to water, that that is a very important factor.

I determine the capacity of the soil to hold water by this way rather than by the use of the funnel recommended in the method of soil analysis, because it is such an uncertain result which you get by pouring the soil onto the funnel and pouring water on it and weighing it after the excess of water has passed off. If you know the weight of the soil and the specific gravity you can find out the amount of space there that water could occupy in the soil. This agrees very closely with actual determinations where the soil spaces are completely filled with water.

Now, as to section 55. It seems to me it is a very crude method, and it is hard to see just what the results mean. Instead of doing this, I have a method here for determining the relation of soils to water. I make an excavation in the field large enough to get into and work easily, usually about two feet square and 18 inches deep. I cut this out with a spade if I have it or with this heavy trowel, which I always carry. Then I cut out a place still further back and remove it to the depth of the top of the subsoil. I clear this shelf off and then with this narrow saw blade and this carving knife I cut out a column of subsoil. Of course the soil can be examined in the same way, but I usually prefer to take the sample just below where the change occurs between the soil and subsoil. I carve out a column of the subsoil exactly 2 inches square and 3½ or 4 inches deep, getting it as nearly square as possible. There is no trouble in maintaining this column of subsoil even with a sandy land. This brass cylinder, 3 inches long and 3¼ inches in diameter, is slipped over the column of subsoil and the space between the subsoil and the sides of the brass cylinder is filled with melted paraffin.

I have a little alcohol stove and a pot of paraffin, and while I am cutting out the column of subsoil the paraffin is melting. I am particular not to use it too hot. The paraffin wants to be just melted, with a little scum on the surface, showing it is cooling. This is to prevent any possibility of its soaking into the soil; but I think there is no trouble about this, because it solidifies as soon as it touches the soil. If it is very warm it is better to pour in a little at a time. As the paraffin cools it is well to keep the upper crust open with a straw or stick until the paraffin has solidified throughout, so that the contraction of the paraffin on cooling shall not pull it away from the soil. In this way I get a sample of the soil in its natural position in the field, 2 inches square and 3 inches deep, which I can experiment on in the field or in the laboratory by passing water or air through, as seems desirable.

Ordinarily it is a little difficult to carry this sample around, and I usually make my determinations out in the field. For this purpose a section of the brass tube 2 inches deep is put on top and secured with a broad rubber band, which makes a water-tight joint. I drive this iron rod down into the soil and put a ring from a retort stand on to support this funnel. The funnel is filled with fine gravel which I have in this bag, and the column of soil embedded in paraffin rests on this gravel. It takes an inappreciable time for the small quantity of water which is used to filter through the gravel, so that this may be neglected in the observation. Water is carefully poured onto the soil, being careful not to disturb the surface of the soil. For this purpose a little fine gravel or coarse sand can be put on the surface of the soil. In pouring in the water be careful to let it fall onto the paraffin. When the whole apparatus gets saturated and water begins to drop from the funnel I put this glass under it and note the time it will take for a quantity of water to pass through the soil which will fill this glass up to the mark on the side with an initial pressure of 2 inches in depth of water. This quantity of water represents an inch in depth of water over the given area (4 square inches) of soil surface. The time required for this quantity of water to pass through the soil is noted, and ordinarily this is all I do, as I can not afford to carry the sample, as I may have to take perhaps eight or ten such samples. I should prefer having them in the laboratory; but when off from the railroad and tramping or driving I can not afford to carry them in if there are many determinations to be made.

This seems a very interesting determination to me. Of course there are some little details that have to be looked after. If you have a stiff clay soil, you can't cut it off top and bottom without smoothing it over and puddling it in a way. You have to look out for that. I have tried breaking it off or sawing it off with this little saw blade, which seems to leave the surface in good condition. Of course it depends upon the nature of the soil. Many of the soils I have been working on are sandy and sandy loams, and I have no trouble of this kind. I find that the method works very nicely. In the same class of soils I get very uniform samples and very concordant results in the flow of water through them. In southern Maryland there are rather light and sandy soils which are very uniform in texture. The wheat-land soils are stronger and have more clay than the lighter truck lands, and it takes a longer time for the water to run through them, but it is quite uniform for samples from different localities in the same soil formations; and so on for the heavier soils until we come to the almost impervious clay lands of the Potomac formation, and with these it takes a good many days for the water to run through.

I have, also, a method here which is very convenient for taking samples of soil for moisture determinations. I was anxious to get a continuous record of soil moisture, but there is no satisfactory way of doing

this, so I wanted to have a determination at least daily. I had a lot of these brass tubes cut off, three-fourths of an inch in diameter and 9 inches long, with a 6-inch mark on the tube. I have correspondents in the different soil formations take a sample of the soil at 8 o'clock every morning. They push the tube down into the soil 6 inches deep and lift it out with the soil contained. I should rather have the samples taken from 3 inches to 9 inches deep, in that way rejecting the top 3 inches, but I see no good way of doing that, as I can not ask too much of the men who voluntarily do this work.

There is but little danger of the soil dropping out, even in light sandy lands. As the tube is withdrawn from the soil one of these rubber finger caps is put on either end, which makes a perfectly airtight joint, and the soil can be kept in this tube for days with no apparent loss from evaporation. The tube with these rubber caps on is put in one of these long bags and sent by mail. I have kept them for a long time and found no loss of weight. The moisture determinations are made in the laboratory then by the usual methods. The tubes hold about 60 to 70 grams of soil.

This is my outfit here just as I take it into the field, and I find it very convenient. In addition, I think the results of this field work are exceedingly interesting. [Applause.]

Mr. HUSTON. I move that the reporter on soils be requested to investigate the Grandeau method in connection with the subject next year.

Adopted.

Mr. DAVIDSON. I move that the alternate method for the determination of chlorin in the ash be left in.

Adopted.

At 5 o'clock p. m. the association took an adjournment to Saturday, August 27, 1892, at 9:30 a. m.

THIRD DAY.

MORNING SESSION, SATURDAY.

The association met at 9:30 a. m. Saturday, August 27, 1892, pursuant to adjournment.

The PRESIDENT. The amount of work we have to get through to-day is so large that I think we had better commence reading the reports and get through with them as soon as possible, and the recommendations can be considered later. The first paper on the program is the "Report on cattle foods low in carbohydrates."

Mr. Jordan's paper on cattle foods low in carbohydrates was read by Mr. Anderson, as Mr. Jordan was unable to attend in consequence of illness.

CATTLE FOODS LOW IN CARBOHYDRATES.

(Read by Mr. ANDERSON.)

The original and doubtless the present main object of the Association of Official Agricultural Chemists is the securing of greater accuracy and uniformity in methods of analysis. The need for the existence of such an association was first shown by the large discrepancies between the results of different chemists working on the same substance. Great improvement has been effected by the adoption of uniform methods of analysis, so that whether the results reached by different analysts tell much or little about the actual composition of a plant, for instance, they are certainly more nearly alike.

It appears, however, that whether we regard the uniformity or the accuracy of the results reached, cattle foods are still in worse plight than either fertilizers or dairy products. The figures reported to this association during the last two or three years support this statement.

The methods of analysis now in use for cattle foods are highly unsatisfactory. All our determinations, when expressed in terms of proximate constituents, represent only approximations which in some instances scarcely approximate. There is serious doubt whether it is worth while for this association to spend any more time in trying to secure uniformity in the determination of what is designated by that meaningless term "ether extract," so long as our analyses of feeding stuffs are chiefly important in the domain of animal nutrition rather than commercially. How much more do we know about a fodder after we have an "extract" which contains fats, wax, chlorophyl, etc., in unknown proportions, whether we consider it either as to its food or its money value?

It is many times more important to know how to separate the vegetable fats in a pure condition than it is to know how to secure a uniform amount of an extract representing nothing in particular, and in our ignorance of how to do the former thing we may just about as well stop trying to do the latter. Your reporter would not be so discourteous as to suppose for a moment that any members of this association do not fully understand how unsatisfactory the methods of analysis for foods now are, and the above remarks are made only for the purpose of helping to arouse the association to greater activity along certain lines.

We now stand face to face with the fact that until there are less unknown compounds in cattle foods, and until we can better identify and separate the known, we shall make but little valuable progress.

As illustrating the sort of work we need there may be mentioned the researches at the Connecticut Experimental Station on the proteids of oats and maize, and the study which Dr. W. E. Stone has been giving to a class of compounds found in the nitrogen-free extract of fodders, which he calls pentosans, in the identification and quantitative determination of which he has made highly creditable progress.

In accordance with the above ideas your reporter very zealously resolved, after the meeting of 1891, that he would direct his efforts as reporter towards securing severe investigations of the facts that are fundamental to certain analytical processes, rather than towards the comparison of different analysts, although such comparison is still needful and fills an important place.

The following circular was sent to quite a number of chemists, the most of whom are especially interested in the analysis of feeding stuffs, and seven replied that they would undertake to study one or more of the points, with some encouragement from others.

Two points, viz, Hönig's method for fiber and starch, and the use of certain oxidants and quartz in ash determinations, were included by vote of the association.

CIRCULAR.

Determination of moisture.—Compare the per cent of loss in weight when the substance is dried according to the official method and when dried in common air for the same time and at the same temperature. It is probable that a loss of organic matter

will occur in either case, and it is desirable to know the relative amount by the two methods.

Ash.—(See Bull. 31, pp. 69, 75.) Compare the official method with the use, separately, of nitric acid, ammonium nitrate, and quartz. Determine the CO_2 and carbon in the ash obtained by several methods compared. Query. Does not the use of nitric acid or ammonium nitrate cause a loss of chlorin.

Ether extract.—Test Patterson's method for the purification of the ether extract. It is obvious that it is necessary to know whether the charcoal retains any fats, and also whether substances not properly classified under the term fats or oils pass through the charcoal. Test the relative effect of the two methods of drying mentioned above upon the per cent of ether extract.

Separation of fiber from starch, etc.—(See Bull. 28, p. 31, and Bull. 31, p. 63.) Test Honig's method for the separation of fiber from starch, etc. (See Fres. Zeit. f. anal. Chem., Vol. 30, p. 91; also Jour. Anal. and App. Chem., 1891, p. 234. For original description of the method see Chem. Zeit., 14, pp. 368, 902.) The method should be tried with a known mixture of cellulose, starch, sugar, and albuminoids in order to ascertain whether the separation is complete and whether the cellulose remains intact. It should also be compared with the official method.

The fiber obtained by Honig's method should be tested for ash and nitrogen. Prof. W. E. Stone suggests that the treatment with glycerin may not remove the pentosans entirely, and it is hoped that Prof. Stone will consent to test samples of fiber from this method for the presence of pentosâns. It will be well to preserve the fiber obtained.

Samples of timothy hay and wheat bran have been sent for use in the above work.

It is not expected that any one chemist will take up all the above points for study, but it is urged that whatever work is done be as exhaustive as possible. Each chemist is expected to largely devise his own methods of studying the points mentioned.

The reports of the work should be in the hands of the reporter by July 1.

It was a disappointment to receive returns from only four chemists, although it is easy to understand why there may have been entirely sufficient reasons for every failure to do work. It was found impossible at the Maine Station to do all that was planned at first. Nevertheless, some interesting results can be reported.

MOISTURE.

In all cases the moisture was determined by two methods, and in two instances by three. The following tables show the figures as received.

Moisture in hay.

	In water or steam oven.		In current of air.		In current of hydrogen.	
	Time.	Per cent.	Time.	Per cent.	Time.	Per cent.
	Hours.		*Hours.*		*Hours.*	
Bartlett and Merrill, Maine	4	6.20	8	7.27	4	7.27
Farrington, Illinois	8	5.92	.....	.....	8	7.65
Krug, U. S. Department of Agriculture	6	6.82	6	6.61	6	6.64
Woll, Wisconsin	5	5.73	.....	.....	.....	6.26

Moisture in bran.

	In water or steam oven.		In current of air.		In current of hydrogen.	
	Time.	Per cent.	Time.	Per cent.	Time.	Per cent.
	Hours.		*Hours.*		*Hours.*	
Bartlett and Merrill, Maine	4	10.24	8	10.71	4	11.70
Farrington, Illinois	8	9.60	.....	.....	8	11.50
Krug, U. S. Department of Agriculture	6	8.87	6	11.13	6	11.11
Woll, Wisconsin	.....	10.20	.....	.....	.....	10.75

Maine.—After passing the air and hydrogen over the substance they were then conducted through sulphuric acid, and a coloration from organic matter was observed with both. Several determinations were made, where the air previously freed from water and CO_2 was passed through weighed sulphuric acid and potash bulbs after leaving the substance (hay). As the increase in weight of the sulphuric-acid bulbs was always greatly larger than the loss in weight of the substance, although no defects appeared in the apparatus, not much definite information was gained. The amount of CO_2 absorbed by the potash bulbs was small in satisfactory determinations not exceeding .005 grams from 2 grams of substance. In one instance the air that had been dried in the apparatus like the one described in Report Connecticut Experiment Station (1889, p. 181) was passed through water for eight hours, but no SO_3 could be found in the water afterwards.

Illinois.—Mr. Farrington's interesting comments on his moisture determinations, which include several samples besides these sent by the reporter, are given here entire:

"A number of samples were dried in ground-glass-stoppered weighing bottles $1\frac{1}{2}$ inches high by 1 inch in diameter. These bottles were used in order to prevent the absorption of moisture while weighing the dried sample after it had cooled in the desiccator. Two grams of each sample was weighed into these bottles, which were heated in a 6 by 8 inch double-walled water oven. A thermometer in this oven registered 98°. Another portion of the same sample was dried in a current of dry hydrogen. This was done by weighing 2 grams into a glass tube 9 inches long and three-eighths inch in diameter (inside). These tubes were then placed in copper tubes of a little larger diameter, passing through a copper box. This box is nearly filled with water, which is heated to boiling. The hydrogen is dried by the apparatus designed by Prof. S. W. Johnson. Ten samples can be dried at one time, each having a separate supply of hydrogen. After passing over the substance in the glass tube the hydrogen bubbles through distilled water. In this water there is accumulated H_2SO_4 and organic matter. Repeated observations have shown that hydrogen passing through sulphuric acid carries the acid (mechanically?) beyond the receptacle in which it is held.

"The following table gives a record of results obtained by drying samples by the two methods described:

Per cent of loss in weight when dried at the temperature of boiling water.

	In hydrogen.				In water oven.			
	4 hours.	8 hours.	16 hours.	24 hours.	4 hours.	8 hours.	16 hours.	24 hours.
Bran from Jordan	11.50	11.80	11.80	.....	9.40	9.70	9.60	.....
	11.20	11.30	11.30	.....	9.30	9.50	9.65	.....
Hay from Jordan	7.40	7.65	7.80	7.95	5.65	6.05	5.95	.....
	7.50	7.65	7.90	8.00	5.45	5.80	5.88	.....
Hay from Illinois	7.80	7.80	.....	.....	6.40	6.65	6.87	.....
	7.80	7.85	.....	.....	6.40	6.65	6.70	.....
Ensilage from Illinois	7.95	8.40	8.95	9.00	6.25	6.90	7.45	7.60
	8.00	8.50	8.85	8.95	6.30	6.45	7.55	7.70
Rock candy (ground)	1.30	1.50	1.65	.....	.95	1.05	1.12	.....
	1.05	1.50	1.55	.....	.95	1.00	1.10	.....
Starch (ground)	7.55	10.85	11.15	.....	8.80	9.00	9.00	.....
	9.35	10.35	10.52	.....	8.95	9.00	8.95	.....

"These results all show a greater loss of weight when heated in hydrogen as compared with the water oven. In nearly every case a constant weight was not reached when the material was repeatedly heated and weighed. This continued loss in weight was not very great, except in the case of ensilage, which lost 1 per cent more in weight by sixteen than by four hours' heating. The loss in weight by starch, when it was heated in hydrogen, was very irregular.

"Sulphuric acid and organic matter were found in the water through which the hydrogen passed after it had passed over the feeding stuffs. The drying apparatus is now provided with an arrangement for passing the dry hydrogen through fused KOH before it reaches the substance to be dried in the oven.

"It is the present opinion of the writer that a more uniform loss of weight or per cent of 'moisture' would be recorded by different chemists working on the same sample if the material was heated in ground-glass-stoppered weighing bottles in an oven surrounded by boiling water than by heating 'four hours in a current of dry hydrogen.' By the latter method the dryness of the hydrogen used may be a variable quantity, and the amount of organic matter contained in the loss of weight is probably different because of the variations that may occur in the volume of hydrogen gas that passes over or through the material heated.

"Weighing the tubes containing the material requires some simple precautions familiar to most chemists. After the tube has been heated in hydrogen the cork may be removed a short time before weighing in order to replace the hydrogen by air. The writer has found this would make an average difference of 0.005 gram in the weight of the tubes used.

"If the fresh substance is weighed into an air-dry tube it (the tube) should be in the same air-dry condition when weighed after heating; otherwise the hydroscopic moisture on the tube itself counts as loss of weight from the substance analyzed."

United States Department of Agriculture.—The figures marked "Moisture in H at 100" were obtained by drying the samples in tubes surrounded by steam for 6 hours, keeping a slow current of hydrogen passing through them. Those marked "in air at 100" were obtained in the same way, except that air was used. Those marked "in air bath at 100" were obtained by drying 6 hours in an ordinary asbestos-jacketed air bath.

Wisconsin.—The plan followed was to weigh out eight samples of each fodder to dry four in hydrogen and four in air at 100. Two of each set were afterwards used for extraction by official method and the other two by the animal charcoal method. Where the single results did not come out satisfactorily, the work was repeated and hence in some cases the results are the average of a large number of analyses.

COMMENTS.

It is to be noticed first, that in general larger percentages of apparent moisture are obtained with a current of either dry air or dry hydrogen than by drying in an ordinary water or steam oven. Similar results have been reached by Jenkins (Bul. 19 A. O. A. C., p. 6) and Snyder (Cornell Agr. Expt. Sta., 1889, Bul. 6). Except in one case figures here noted show no large differences between the currents of dry air and dry hydrogen. The same was also true in Snyder's work. There are at least two possible explanations of the larger percentages of moisture where a current of dry gas is used, as compared with the old method.

(1) A greater actual loss of moisture.

(2) Greater volatilization of organic material with a current of gas. That the loss in weight by drying in a current of gas is to some slight degree, at least, due to volatile organic compounds is generally understood, but the extent of this loss, whether greater or less than in the common water oven, is not known. We would expect it to be greater, however.

Mr. Farrington's observations, if generally true, that H_2SO_4 is carried mechanically through the substance by the current of gas, might explain in part a loss of com-

pounds other than moisture. But this transferrence of H_2SO_4 did not occur at the Maine station. In 1885 Morley passed from 7,000 to 8,000 liters of air through a Winkler absorption tube containing sulphuric acid of a sp. gr. 1.834, and this volume of air took up only from 2.5 to 3.1 mg. of SO_3, whether as gaseous sulphur trioxid or as H_2SO_4 mechanically. (See Fres. Zeit. anal. Chem., 1885, p. 533.) From this there would not appear to be any especial danger pertaining to H_2SO_4 as a means of securing dry air or hydrogen.

Greater uniformity of results is noticed when a current of hydrogen was used than by the ordinary air drying, but even with the hydrogen this agreement is not at all close.

ASH.

In three laboratories comparisons of the official method have been made with the use of such oxidants as nitric acid and ammonium nitrate, also with quartz.

A summary of the results that may properly be compared is given below.

Ash.

	Crude ash.	Ash corrected for CO_2 and C.	Ash by official method.	Ash using HNO_3.	Ash using $(NH_4)NO_3$.	Ash using quartz.
Timothy hay:	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Bartlett, Maine	.....	.....	5.20	4.95	5.00	5.23
Farrington, Illinois	.....	.....	5.32	.....	.....	.....
Krug, United States Department of Agriculture	.....	.....	4.89	5.57	5.55	2.53
Woll, Wisconsin	5.25	4.55	5.19	4.71	5.04	4.65
Wheat bran:						
Bartlett, Maine	.....	.....	5.47	5.30	5.27	.....
Farrington, Illinois	.....	.....	5.20	.....	.....	.....
Krug, United States Department of Agriculture	.....	.....	5.43	5.46	5.45	3 70
Woll, Wisconsin	5.68	5.36	5.45	5.17	5.23	5.29

NOTES.

Maine.—The official and the quartz methods were much the most satisfactory, as the combustion with the nitric acid or ammonium nitrate was so violent, even when the substance was previously charred, as to render special precautions necessary in order to prevent loss.

Illinois.—No loss in weight was observed when Na Cl and KCl were heated in a platinum dish on asbestos paper or in a porcelain dish over No. 60 wire gauze over the full heat of a Bunsen burner.

The sample of bran did not contain any chlorin. The pure ash was estimated in both samples, hay and bran, by charring, boiling with water, filtering, igniting residue, and to it adding the water extract. This sum was taken as the standard with which the per cents of ash obtained in other ways were compared. Hay 5.32 per cent, bran 5.20 per cent. The hay burnt to a constant weight and white ash in a platinum dish on asbestos paper. This ash 5.35 per cent. The bran could only be burnt white by heating to a full red heat. This ash, 5.15 per cent.

The use of ammonium nitrate, nitric acid, or quartz sand, seemed to the writer more objectionable than extracting the chlorids, if any, with water, and igniting the residue.

United States Department of Agriculture.—Ash was done, firstly, by the official method; secondly, by partly charring, moistening with nitric acid and reburning (marked

HNO_3), thirdly, by partly charring, mixing with solid ammonium nitrate (marked NH_4NO_3); and, finally, by mixing the feeding stuff with kieselguhr (diatomaceous silica), using one gram of each, and burning (marked kieselguhr).

Wisconsin.—About two grams of material were ignited in muffle oven until the ash was grayish white. In many cases the porcelain dishes did not lose any in weight on renewed heating in muffle oven.

This is crude ash in the following. Some samples were ignited over direct flame; first at a low heat, and afterwards over blast lamp. To other samples ammonium nitrate solution or concentrated nitric acid was added after crude ash had been determined. These samples were then carefully dried, heated at a low red heat, and afterwards ignited over blast lamp. Chlorin was determined in the crude ash, and the ash from nitric acid, ammonium nitrate and quartz methods using one-fortieth normal silver nitrate solution with potassium chromate as indicator. The results were corrected for the quantity of chlorin in the chromate solution.*

	Per cent.
A. Crude ash:	
Sample 1 (14 analyses)	5.25
Sample 2 (12 analyses)	5.68
B. Official method:	
Sample 1 (4 analyses)	5.19
Sample 2 (2 analyses)	5.45
Corrected for carbonic acid and charcoal:	
Sample 1	4.55
Sample 2	5.36
C. Direct ignition:	
Low red heat:	
Sample 1 (2 analyses)	4.98
Sample 2 (2 analyses)	5.17
Blast lamp:	
Sample 1 (2 analyses)	4.81
Sample 2 (2 analyses)	5.15
Corrected for carbonic acid and charcoal:	
Sample 1	4.44
Sample 2	5.06
D. Ammonium nitrate:	
Low red heat:	
Sample 1 (2 analyses)	4.71
Sample 2 (4 analyses)	5.23
Blast lamp:	
Sample 1 (2 analyses)	4.56
Sample 2 (2 analyses)	5.13
E. Nitric acid:	
Low red heat:	
Sample 1 (4 analyses)	5.04
Sample 2 (4 analyses)	5.17
Blast lamp:	
Sample 1 (4 analyses)	4.77
Sample 2 (2 analyses)	5.17
F. Quartz sand:	
Low red heat:	
Sample 1 (2 analyses)	4.65
Sample 2 (4 analyses)	5.29

*Mr. Woll writes that in his determinations by the official method he did not ignite at a low red heat after adding the soluble parts of the ash to the more strongly ignited portion, but only dried at 110°. The percentages would probably be lower if ignition had been performed.

F. Quartz sand—Continued.

	Per cent.
Blast lamp:	
Sample 1 (2 analyses)	4.59
Sample 2 (4 analyses)	5.10

Chlorin in ash.

Crude ash:	
Sample 1 (2 analyses)	1.46
Sample 2 (2 analyses)	.15
Quartz sand, blast:	
Sample 1 (2 analyses)	1.44
Sample 2 (2 analyses)	.13
Ammonium nitrate:	
Red heat: Sample 2 (2 analyses)	.05
Blast:	
Sample 1 (3 analyses)	.04
Sample 2 (2 analyses)	.03
Nitric acid:	
Red heat:	
Sample 1 (2 analyses)	.08
Sample 2 (2 analyses)	.01
Blast lamp:	
Sample 1 (4 analyses)	.11
Sample 2 (2 analyses)	.01

COMMENTS.

It is worthy of remark that with the hay the use of HNO_3 or $(NH_4)NO_3$ gave ash considerably higher than was obtained when the crude ash is freed from CO_2 and C, and from the United States Department laboratory the figures are nearly .66 per cent higher than by the official method. In the case of the bran the different methods gave more nearly uniform results all around. This is what we should expect. Any ash containing the alkalis largely combined with CO_2 or chlorin, would be materially modified in composition by the use of HNO_3 or NH_4NO_3, the chlorids and carbonates of the alkalis being converted to nitrates which by ignition would be changed wholly or largely to nitrites. As the nitrites have a higher molecular weight than the oxids or even the carbonates of the alkalis their formation would materially increase the percentage of ash. For instance, take Hay No. CCXL, the ash of which was analyzed at the Connecticut Experiment Station (*see* Rep., 1886, p. 118) and was found to contain 34.4 per cent of potash and 0.77 per cent of soda. Allowing that all the SO_3 present would combine with the alkalis and the P_2O_5 with other bases, the use of HNO_3 or $(NH_4)NO_3$ would theoretically give an ash over 1 per cent higher than the true ash, provided the latter be regarded as the crude ash minus CO_2 and C. In the case of the cereal grains, however, where the acids less volatile than HNO_3 are practically equivalent to the bases, no such changes would occur, and if the use of these oxidants facilitates the burning, they certainly appear to be admissible. The very low percentages reported by Mr. Krug with the use of kieselguhr (diatomaceous silica) are not easy to understand, but it is fair to suppose that the silica was in a form which enabled it to decompose the carbonates, sulphates, and chlorids, with the formation of silicates, which would in part explain the very large differences observed.

CRUDE FIBER.

It is greatly to be regretted that Hönig's method for fiber and starch was tested in only one laboratory.

Mr. Merrill in consultation with myself has given the method all the study which time permitted, although the work was not carried far enough to reach any conclusion as to the value of the process with feeding stuffs.

The details of the Hönig method as published are briefly as follows (see Fres. Zeit., anal. Chem., p. 91): 2 grams of substance are heated from one-half to three-quarters of an hour in 60 cc. of glycerol at a temperature of 210°, with frequent stirring, the tube containing the glycerol being immersed in a sulphuric-acid bath. The author states that at 150° the glycerol begins to froth because of the escaping vapor of water. As a rule the foaming ceases at 190°. Care should be taken to bring into the glycerol again the portion of the substance which becomes attached to the sides of the tube above the liquid. After treatment for thirty to forty-five minutes the glycerol solution is allowed to cool to 130°, and is then turned in a small stream into 200 cc. of 95 per cent alcohol, which is stirred at the same time. The tube and thermometer are washed off with hot water, not over 50 cc. being necessary.

The alcoholic solution, diluted with the wash water, is completely cooled, filtered through a plaited filter, and the residue washed with a mixture of 5 parts alcohol and 1 part ether. In order to remove the greater part of the alcohol and ether the residue in the filter is placed on a porous clay plate. The residue is then washed into a beaker with 100 to 150 cc. hot water, and is heated over a flame or steam bath until the alcohol is all removed. The remaining fiber is thrown upon a tared filter, washed with hot water, and dried at 110° and weighed. The author states that the last filtration is rendered less difficult if 10 cc. HCl (sp. gr. 1.125) are added to the liquid previously and then heated on a water bath for a half hour. (The statement is made that the cellulose is not affected by this treatment with HCl, but this should have proof. The author also states that the fiber thus obtained contains much ash and but little nitrogen, but as he mentioned 1 per cent as the maximum per cent of nitrogen found, which would be equal to 6.25 per cent albuminoids, his statement seems somewhat peculiar.) The filtrate from the fiber is brought to a volume of 250 cc., to 200 cc. of which are added 12 cc. HCl, sp. gr. 1.125. The whole is heated on a water bath for two to three hours, and the sugar in the solution is determined in the ordinary manner.

Gabriel has given Hönig's method a trial (see Zeit. phys. Chem., XVI, p. 370), and he found that it gives results much higher than the Weende method. He also found that a large percentage of ash and nitrogen were left in the fiber from meadow hay and from the seed of golden lupine. One gram fibrin nearly but not entirely dissolved in 60 cc. glycerol at 210°, but on addition of water or alcohol a flocculent precipitate formed.

The author suggests a modification of Hönig's method, as follows: He adds to the glycerol 2 grams KHO, heats the glycerol solution only to 180°, cools to 140°, turns it into 200 cc. boiling water, allows the residue to settle, decants through a linen filter, boils the residue twice more in 200 cc. water, the last time adding 25 cc. of 25 per cent HCl. This modification gave results with quite a list of substances not greatly different from the Weende method.

The author made some experiments with modified glycerol-potash method, which he seems to regard as showing it to be an improvement on the original method.

Fibrin, egg albumen, conglutin, etc., were easily and completely dissolved, and no preciptate was formed by the addition of alcohol or water.

When 2 grams Swedish filter paper were treated by the glycerol-potash method a loss of 8.63 per cent occurred. When the residue from the filter paper was boiled with 200 cc. 0.6 per cent HCl solution no further loss took place. The author states that without doubt fiber which is separated by the glycerol-potash method still contains nitrogen-free substance which he is equally certain will be inverted by boiling with very dilute acid without affecting the fiber, though it is difficult to discover that this offers sufficient experimental proof of these opinions.

Quite an amount of time has been spent at the Maine Station in studying Hönig's method. Some difficulties were encountered which led to a few modifications. Paraffin was substituted for the sulphuric-acid bath, a vessel of agate ware, of about 800 cc. capacity being used in which to heat it.

The digestions were made in large test tubes having an internal diameter of 1¼ inches and a length of 12 inches.

Both the glycerol and the fodder contained moisture which was given off on heating, causing the troublesome frothing mentioned in the original description of the method. This difficulty was avoided by drying the fodder before using and by boiling the glycerol in an open porcelain dish until the water was practically all driven off. Scarcely any frothing then occurred. Hönig's and the official method were compared, using the hay and wheat bran, with results as below:

Crude fiber.

Hönig's method.		Official method.	
Bran.	Hay.	Bran.	Hay.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
12.92	39.20	6.47	27.40
12.76	38.46	6.43	27.45
12.36	40.37	6.86	27.62
12.32	40.09	6.59	27.86
13.35	40.32	6.42	27.71
12.70	39.08	6.50	27.65
.....	38.55	.....	.....
.....	38.60	.....	.....
Average, 12.73	39.33	6.54	27.61

	Hönig's method.		Official method.	
	Hay.	Bran.	Hay.	Bran.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Residue after extraction	39.330	12.73	27.61	6.54
Nitrogen in residue	.2530	.23	.09	.06
Ash in residue	1.670	1.75	.53	1.79
Fiber corrected for nitrogen and ash	38.060	12.38	27.30	6.40

The two methods were tried on mixtures of fiber, starch, and sugar to determine their relative effect on the fiber. The fiber was prepared by treating a fine sample of cotton with very dilute HCl, bromin water, dilute ammonia, alcohol, and ether.

	Hönig's method.		Official method.	
	1.	2.	1.	2.
Mixture taken:				
Fiber	.4040	.4330	.4043	.4692
Starch	.9000	.9000	.9000	.9000
Sugar	.0500	.0500	.0500	.0500
Fiber recovered	.4038	.4339	.3628	.3935
Per cent loss	None ...	None ...	10.2000	16.1000

Our own work substantiates at least part of Gabriel's conclusions. The experiments so far made seem to warrant the following remarks:

(1) Fiber is not affected by treatment with glycerol at 210°.

(2) The residues from the glycerol treatment contain more ash and nitrogen than those from the official method, although much less of these were found in the Hönig fiber at the Maine station than in Gabriel's experiments.

(3) A much larger residue is left when feeding stuffs are treated by Hönig's method than when the official (Weende) method is used, even after allowance is made for the nitrogen compounds and ash.

(4) Part of the difference in the two methods may be accounted for by Prof. Stone's suggestion that the glycerol treatment would not wholly remove the pentosans. At the same time the solutions of acid and alkali used in the Weende method confessedly exert no small solvent action upon cellulose, which would explain to some extent the lower figures by the old process.

(5) Gabriel's glycerol-potash modification does not appear to be an improvement over treatment with dilute acid and alkali, inasmuch as the glycerol-potash solution acted upon filter paper, and when applied to feeding stuffs left a residue no larger than from the Weende method, where the solution of cellulose is acknowledged.

(6) Hönig's method is certainly sufficiently promising to make it worthy of further study.

ETHER EXTRACT.

The following figures were reported by the several analysts for ether extract:

Ether extract in hay.

	Air-dry material.	After drying—		Patterson's charcoal method.
		In air.	In hydrogen.	
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Farrington, Illinois	3.25	3.00	2.75	.....
Krug, United States Department of Agriculture	.....	.....	2.61	.....
Patterson, Maryland	5.04	4.30	.....	[1] 1.43
Woll, Wisconsin	.....	2;61	2.74	[2] 2.34

Ether extract in bran.

	Air-dry material.	After drying—		Patterson's charcoal method.
		In air.	In hydrogen.	
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Farrington, Illinois	4.70	4.50	4.37	.....
Krug, United States Department of Agriculture	.....	.....	4.03	.....
Patterson, Maryland	4.87	4.55	.....	[1] 3.30
Woll, Wisconsin	.....	4.34	3.70	[2] 3.59

[1] Substance previously dried two hours in water oven.
[2] Substance previously dried in hydrogen.

NOTES.

Illinois.—The differences in per cents of ether extract of the air, hydrogen, or oven dry material varied with the substances extracted. The following results were obtained by extracting 2 grams for sixteen hours in a Soxhlet apparatus with absolute ether, which, before using, had been digested with $CaCl_2$.

Per cent of ether extract.

[The extractions went at the rate of nearly 20 siphonings per hour.]

	Material dried before extraction in—		Air-dry material.
	Hydrogen.	Water oven.	
	Per cent.	*Per cent.*	*Per cent.*
Bran from Jordan	4.3–4.45	4.50	4.70–4.75
Hay from Jordan	2.75	3.00	3.25–3.25
Hay from Illinois	2.85	.....	3.05
Ensilage from Illinois	1.38–1.45	1.75–1.35	2.32–2.30

One sample of hay gave about the same per cent of ether extract from the air as from the hydrogen-dry sample. The other hays and the ensilage did not. The ether extract of the air-dry substance was about 1 per cent more than on the hydrogen-dry sample. A few determinations of nitrogen were made in the air-dry substance, also on the residue after drying in hydrogen and extracting with ether.

Per cent of nitrogen.

	Air-dry substance.	Residue from drying in hydrogen and ether extraction.
	Per cent.	*Per cent.*
Hay	0.74	0.81
Bran	2.70	2.40
Ensilage	1.18	0.97
Corn meal	1.61	1.65
Corn and cob meal	1.54	1.57
Oil meal	5.30	5.20
Oats	2.22	2.21
Cheese[1]	4.13	4.19

[1] From ether extraction only.

Wisconsin.—By mistake, only 1 gram of charcoal was taken in the first determination of ether extract in sample 1, and 2 grams in the case of sample 2. In repeating the determination I had a slight explosion in the hydrogen oven which destroyed all the material which was left of sample 1, so my figures for the animal charcoal method with this sample are not comparable with others found where a larger quantity of charcoal was used.

Results of analyses.

	Per cent.
Air drying, official method:	
Sample 1 (4 analyses)	2.61
Sample 2 (4 analyses)	4.34
Air drying, animal charcoal:	
Sample 1, 1 gram (2 analyses)	2.80
Sample 2, 4 grams (4 analyses)	3.83
Dried in hydrogen, official method:	
Sample 1 (2 analyses)	2.74
Sample 2 (4 analyses)	3.70
Charcoal method:	
Sample 1, 2 grams (2 analyses)	2.34
Sample 2, 4 grams (4 analyses)	3.59

Ether extract, indirect:

Air drying, official method:	
Sample 1 (4 analyses)	2.71
Sample 2 (4 analyses)	4.97
Charcoal method:	
Sample 1, 1 gram (2 analyses)	2.72
Sample 2, 4 grams (4 analyses)	3.20
Dried in hydrogen, official method:	
Sample 1 (1 analysis)	3.04
Sample 2 (4 analyses)	5.08
Charcoal:	
Sample 1, 2 grams (2 analyses)	3.00
Sample 2, 4 grams (2 analyses)	4.95

The extraction was continued for eight hours, the extract then dried and weighed and the extraction continued for eight hours more, in order to study if, possibly, by the charcoal method extraction was complete in a shorter time than by the official method.

Results of eight hours' extraction—Averages of two analyses.

	Per cent.
Dried in air, no charcoal:	
Sample 1	2.38
Sample 2	4.01
Charcoal:	
Sample 1	2.43
Sample 2	3.68
Hydrogen, official method:	
Sample 1	2.46
Sample 2	4.02
Charcoal:	
Sample 1	2.04
Sample 2	3.24

The ether extracts by the animal charcoal method, in case of both samples, were light colored, and to all appearances purer fat than by official method. In case of the timothy hay the extract in the charcoal method was still green colored, showing that all the chlorophyl had not been taken up by the charcoal. In order to obtain some definite idea in regard to the purity of the extract, Koettstorfer's method was applied to the extract. A good deal of work was expended in trying to get satisfactory duplicates, but finally by the use of a seminormal alcoholic soda solution made up from a saturated solution of soda, and by using alcohol distilled over calcium carbonate, good results were obtained, although in some cases duplicates might still vary quite a little owing to the small amount of the extract. The results are figured on the quantity of potash taken up by 1 gram of fat, this being the more ordinary method.

Results by Koettstorfer's method.

One gram of extract was found equivalent to the following quantities of potash:

	Milligrams.
Dried in air, official method:	
Sample 1 (3 analyses)	115.5
Sample 2 (4 analyses)	185.9
Charcoal:	
Sample 1, 1 gram (2 analyses)	165.0
Sample 2 (3 analyses)	222.1

	Milligrams.
Dried in hydrogen, official method:	
Sample 1 (1 analysis)	135.0
Sample 2 (3 analyses)	185.4
Charcoal:	
Sample 1, 2 grams (2 analyses)	149.3
Sample 2 (3 analyses)	226.5

In every case you will notice the ether extract purified by the animal charcoal gave a higher saponification equivalent, although in case of Sample 1 the quantity of charcoal used was evidently insufficient to take out all the chlorophyl. The figures for waxy substances given by König in his Unters. Landw. wicht. Stoffe (page 413), all come at 100 or slightly above, while the oils all come toward 200.

By the Koettstorfer method the butter fat used in subsequent determinations gave 0.2284, the olive oil 0.1894, and the cotton-seed oil 0.1895 grams KOH, for 1 gram of fat.

I have made a large number of analyses to test in how far any fat is retained by the charcoal. The earlier analyses are excluded because the fat was dried in the air after extraction, and those given are to the best of my knowledge correct results, all precautions having been taken that I can think of to secure such. The fats were absorbed by a small piece of cotton and extracted by anhydrous and alcohol free ether, about 4 grams of charcoal being placed at the bottom of the extraction tube; extract was dried in H at 100° till constant weight. Both charcoal and cotton had been extracted for at least thirty-two hours previous to the test.

	Weighed out.	Regained.	Difference.
	Grams.	*Grams.*	*Grams.*
Butter fat	1.2138	1.2119	—.0019
	1.1321	1.1337	+.0016
	.1292	.1233	—.0059
	.2154	.2107	—.0047
	.0946	.0901	—.0045
	.0499	.0415	—.0084
Olive oil	1.0514	1.0507	—.0007
	1.0136	1.0158	+.0022
	.1163	.1160	—.0003
	.2602	.2554	—.0048
	.1016	.0917	—.0099
	.2110	.1975	—.0135
Cotton-seed oil	.0721	.0622	—.0099
	.1068	.0964	—.0104
	.1189	.1108	—.0081

I do not see how I can escape the conclusion, from these results, that the charcoal retains some of the fat. In view of the perfect agreement of Mr. Patterson's results I am greatly puzzled at this, and have looked for mistakes everywhere, without, however, having been able to account for the differences.

COMMENTS.

The ether extract appears to be uniformly less when the substance is dried, than when extracted in an air-dry condition. Moreover the extract was less, except in a single instance, when the substance is previously dried in hydrogen than when dried in air. This appears to be rather odd when it is claimed that hydrogen is the proper medium for drying because air so oxidizes the fats as to render them insoluble in ether.

It is to be noticed that where drying in air and in hydrogen have been compared, the air drying has been done in a water oven, while the hydrogen has been passed over the substance in a current. May it not be possible that a larger volatilization

of ether-soluble compounds occur when the drying medium is applied as a current? Or what shall we offer as an explanation of the results here reported?

We see that in these instances, the ether extracts from the air-dry materials are the largest, and are we sure that drying in any medium at 100° is not prejudicial to a true estimation of the fats. Simply getting a less per cent by drying proves nothing. It is somewhat difficult to understand how 6 or 7 or even 10 per cent of moisture in 2 grams of hay could hydrate the ether sufficiently to cause the solution of compounds that would not be taken out if the 0.140 to 0.200 grams of water were not present.

Do we not need to investigate the ether extract by methods akin to Mr. Woll's determination of the potash equivalent in the several cases, in order to know under what conditions we extract the largest quantity of the true fats.

Mr. Patterson's and Mr. Woll's figures with the use of charcoal differ very widely, with no explanation for the difference unless it be that in any case Mr. Woll used too little charcoal.

Mr. Woll also finds a loss in passing pure fats through charcoal, which is contrary to Mr. Patterson's conclusions.

The PRESIDENT. If there is no objection, we will now have the paper read on food and feeding stuffs low in carbohydrates.

Mr. Anderson presented the following paper on that subject:

(NOTE BY EDITOR.—Mr. Jordan, who was appointed reporter on foods rich in carbohydrates, by some misunderstanding, took for his theme foods low in carbohydrates. There are, therefore, two reports on hay, fodder, etc., and none on cereal foods.)

REPORT ON FOODS AND FEEDING STUFFS LOW IN CARBOHYDRATES.

By J. T. ANDERSON.

That the reporter on "foods and feeding stuffs low in carbohydrates" should have selected timothy hay and corn fodder as subjects for investigation, may require a word of explanation.

A careful study of the discussions which led up to the division of cattle foods convinced me that it was the desire and intent of the association to divide the subject into grains and coarse fodders. The president of the association two years ago, Prof. Scovell, recommended in his address "that the subject of cattle foods be divided into fodder analysis and grain analysis."

In the methods of analysis adopted by the association that same year, coarse fodders are specifically designated as belonging to the class of nonstarchy foods. Strictly speaking, neither timothy hay nor corn fodder could hardly be classed as "feeding stuffs low in carbohydrates," but they are coarse fodders, and for that reason were selected by this reporter. This much by way of explanation seems to be necessary, inasmuch as the other reporter, I understand, has selected timothy hay as one of his subjects.

In the preparation of these samples it need only be stated that, as to grinding, sifting, and mixing, the processes recommended by Dr. Caldwell two years ago, and adopted by the reporter last year, were strictly followed. The samples which were sent to the several analysts were collected in glass bottles and sealed all at the same time, so that they would likely be in the same condition when opened for analysis. Sixteen analysts responded favorably to the request to participate in the work on these samples, though only twelve have thus far reported their work.

The following tables contain the results of the analyses reported (calculated, as is customary, to the dry substance), together with some comparative data which it will be interesting to study:

SAMPLE 1.—*Timothy hay.*

State.	Analyst.	Moisture.	In the dry substance.						
			Ash.	Ether extract.	Crude fiber.	N-free extract.	Crude protein.	Nitrogen.	
								Total.	Alb.
New Jersey	L. A. Voorhees	11.25	6.16	2.48	34.88	50.54	5.94	0.940	0.040
Illinois	E. H. Farrington	[1] 12.75	5.89	2.41	37.61	47.65	[1] 6.44	1,030	.....
Michigan	R. C. Kedzie	[1] 12.45	6.16	2.47	36.00	49.53	5.84	0.935	0.880
Texas	Duncan Adriance	11.31	5.88	[1] 3.47	37.40	46.90	[1] 6.35	1.020	.....
Wisconsin	F. W. Woll	11.91	5.95	[1] 1.68	35.40	51.58	5.39	0.862	0.874
U. S. Department of Agriculture.	W. H. Krug and T. C. Trescot.	11.80	6.34	[1] 4.28	34.52	49.19	5.67	0.907	.....
New Hampshire	C. L. Parsons	11.70	5.66	2.14	[2] 31.88	55.32	5.00	0.800	.....
North Carolina	B. W. Kilgore	11.55	5.92	2.57	34.38	51.14	5.96	0.955	0.920
South Dakota	J. H. Shepard and C. G. Hopkins.	11.61	5.78	2.31	[1] 39.33	47.42	5.16	0.820	0.810
Pennsylvania	J. W. Fields	[1] 10.82	6.10	2.15	34.55	51.38	5.82	0.931	0.919
Kansas	J. T. Willard	11.28	5.92	2.10	36.19	50.27	5.61	0.898	0.832
Alabama	J. T. Anderson	11.43	5.99	2.42	34.58	51.22	5.80	0.929	0.903
Averages		11.54	5.98	2.33	35.55	50.18	5.62	0.919	0.885
Difference between the highest and lowest per cent		.66	.68	.56	3.23	.....	.96	.....	.....
This difference expressed in per cent. of average		5.72	11.37	24.03	9.09	.....	17.08	.....	.....
Widest difference from average		.37	.36	.32	2.06	.....	.62	.....	.....
This difference expressed in per cent. of average		3.20	6.02	13.73	5.79	.....	11.03	.....	.....

SAMPLE 2.—*Corn fodder.*

State.	Analyst.	Moisture.	In the dry substance.						
			Ash.	Ether extract.	Crude fiber.	N-free extract.	Crude protein.	Nitrogen.	
								Total.	Alb.
New Jersey	L. A. Voorhees	11.76	8.44	2.82	27.14	54.45	7.15	1.140	1.140
Illinois	E. H. Farrington	[1] 13.46	7.80	2.37	[1] 29.56	52.47	7.80	1.240	.....
Michigan	R. C. Kedzie	12.39	8.57	2.55	28.36	53.68	6.84	1.094	0.931
Texas	Duncan Adriance	12.28	7.92	[1] 3.62	[1] 29.03	51.21	[1] 8.22	1.320	.....
Wisconsin	F. W. Woll	12.05	8.11	2.25	27.34	54.84	7.46	1.190	0.989
U. S. Department of Agriculture.	W. H. Krug and T. C. Trescot.	12.25	8.06	2.30	27.38	55.95	7.31	1.170	.....
New Hampshire	C. L. Parsons	12.20	7.63	2.38	[2] 25.20	57.50	7.29	1.170	0.832
North Carolina	B. W. Kilgore	12.44	7.08	2.56	27.79	55.36	7.21	1.150	0.954
South Dakota	J. H. Shepard and C. G. Hopkins.	11.18	7.18	2.52	[1] 30.85	52.88	6.57	1.050	0.930
Pennsylvania	J. W. Fields	[1] 11.11	8.26	2.56	27.42	54.39	7.38	1.181	1.091
Alabama	J. T. Anderson	12.11	8.32	2.61	27.22	54.68	7.17	1.147	0.972
Averages		12.14	7.85	2.49	27.59	54.51	7.22	1.168	0.980
Difference between the highest and lowest per cent		.68	1.51	.57	1.22	.....	1.23	.....	.....
This difference expressed in per cent. of average		5.60	19.24	22.89	4.42	.....	17.03	.....	.....
Widest difference from average		.38	.79	.33	.77	.....	.65	.....	.....
This difference expressed in per cent. of average		3.13	10.06	13.25	2.79	.....	9.00	.....	.....

[1] Not included in the estimates.

[2] Used 2½ per cent solutions.

For the purpose of comparing the work of this year with the work of previous years the following table has been prepared. To give greater value to the comparison hay was the substance selected for the comparison in all cases:

TABLE 3.

	Year.	Ash.	Ether extract.	Crude fiber.	Crude protein.
Difference between the highest and lowest, expressed		*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
in per cent of average	1889	.....	96.79	13.47	24.23
	1890	29.59	80.60	27.20	20.70
	1892	11.37	24.03	9.09	17.08
Widest difference from average in per cent of average	1889	.....	48.53	8.75	14.41
	1890	14.80	49.59	13 90	13.80
	1892	6.02	13.73	5.79	11.03

From this table it will be seen that we are making commendable progress in the analysis of coarse fodders, though it must be admitted that there is room for great improvement yet.

REMARKS AND SUPPLEMENTARY INVESTIGATIONS BY THE SEVERAL ANALYSTS.

MOISTURE.

New Jersey.—The samples dried five hours instead of four; gave 11.49 per cent and 11.99 per cent, respectively.

Wisconsin.—The moisture determinations were made in small glass dishes about $1\frac{1}{4}$ inches in diameter and 1 inch high. The copper oven used for drying in hydrogen does not work very satisfactorily.

U. S. Department of Agriculture.—In the following table the figures marked: "In H at 100°," were obtained by drying the samples in tubes surrounded by steam for 6 hours, keeping a slow current of hydrogen passing through them. Those marked "In air at 100°," were obtained in the same way, except that air was used instead of H. Those marked "In air bath at 100°" were obtained by drying six hours in an ordinary asbestos-jacketed air bath.

Water determinations.

[W. H. Krug.]

	In H at 100°.	In air at 100°.	In air bath at 100°.
	Per cent.	*Per cent.*	*Per cent.*
Timothy hay	11.77	12.00	11.45
	11.83	11.99	11.44
Mean	11.80	11.995	11.445
Corn fodder	12.21	12.36	12.04
	12.29	12.39	12.05
Mean	12.25	12.375	12.045

Pennsylvania.—Dried in watch glasses in a common air bath at 100° for six hours.

Kansas.—A current of pure dry hydrogen was passed through the substance. A further drying of five hours showed only a slight additional loss.

ASH.

Wisconsin.—

Ash determinations.

[F. W. Woll.]

Sample.	Official method.	Crude-ash method (in muffle to constant weight).	Latter corrected for CO_2 and char.
	Per cent.	*Per cent.*	*Per cent.*
1	5.23	5.32	4.45
2	7.13	7.27	6.38

Last results more correct than those obtained by the official method. Was unable in any of my work to obtain a white ash by heating at low redness, as required by the official method.

U. S. Department of Agriculture.—Ash was done, first, by the official method; secondly, by partly charring, moistening with nitric acid, and reburning (marked HNO_3); thirdly, by partly charring, mixing with solid ammonium nitrate, and burning (marked NH_4NO_3), and finally by mixing the feed stuff with kieselguhr (diatomaceous sand) and burning.

Ash determinations.

[W. H. Krug.]

	Official.	HNO_3.	NH_4NO_3.	Kieselguhr.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Hay	5.60	5.720	5.685	2.805
Fodder	6.20	7.395	7.350	4.265

South Dakota.—Determinations made by burning in a porcelain crucible over an ordinary gasoline Bunsen burner. Required four to six hours to make the incineration. The crucible was kept at a scarcely perceptible red heat.

Pennsylvania.—Burned to whiteness in platinum dish, heated below redness.

Kansas.—Conducted in good-sized platinum dishes, extraction after charring being employed.

ETHER EXTRACT.

New Jersey.—Samples extracted twelve hours instead of sixteen gave 2.14 per cent, and 2.41 per cent with a second extract (extracting four hours more) of 0.02 per cent and 0.08 per cent, respectively.

Illinois.—By extracting the air-dry samples just as they were received, the ether extract of each sample was just 1 per cent higher than that given on the hydrogen-dried substances.

Michigan.—The ether extract of sample 1, by Patterson's method, gave 2.16 per cent.

Texas.—The extracts were all dried in air instead of in hydrogen.

U. S. Department of Agriculture.—The ether was washed with water, then dried over sodium hydrate, and finally over metallic sodium. The extraction was made in a Knorr extraction apparatus. During the process of extraction the upper end of the condenser was closed by inverting a test tube over it to exclude extraneous moisture.

South Dakota.—Extraction continued actively for sixteen hours in a siphoning apparatus, occupying a part of three days and standing over two nights. This length of time is not considered adequate to thorough extraction.

Pennsylvania.—Extraction done under three conditions, as follows:

Sample.	14 hours without drying.	14 hours after drying 6 hours in air at 100°.	12 hours after drying 4 hours in CO_2.
	Per cent.	*Per cent.*	*Per cent.*
1	3.24	.....	1.92
2	3.68	2.58	2.27

Maryland.—

Fat determinations.

[H. J. Patterson.]

	Official method.		Patterson's modification.	
	In substance as received.	After drying.	In substance as received.	After drying.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Timothy hay	2.63	1.88	1.13	1.03
Corn fodder	3.03	2.33	1.28	.83

Dried for two hours in a water oven before putting into the extractor. Extract dried in the same way for thirty minutes.

Kansas.—The fat was extracted in a glass siphoning apparatus. Absolute ether kept over metallic sodium was used, and the ends of the condensers closed with calcium chlorid tubes. A second extraction with fresh ether for seven hours gave an additional 0.08 per cent of fat for the hay.

CRUDE FIBER.

Wisconsin.—A linen filter was used for filtration after boiling with the acid, suction being employed.

New Hampshire.—Employed 2½ per cent solutions both of acid and alkali, and filtered both times through linen, finally washing into a Gooch crucible. Duplicates excellent.

Pennsylvania.—No condenser was used, but only a little evaporation occurred.

Kansas.—Filtered, after boiling with acid, through a linen filter, whose apex was charged with an asbestos plug.

Texas.—No return-flow condenser used.

NITROGEN.

Michigan.—Used 0.7 grams of the substance and 0.6 grams of copper hydrate and 35 cc. potassium sulphid.

Wisconsin.—No zinc used in the distillation.

Pennsylvania.—Used the Kjeldahl method with mercury, but without potassium permanganate.

Kansas.—Armsby and Short's tube was used to arrest the alkali during distillation, and a current of steam from ammonia-free water was injected to prevent bumping and to hasten distillation.

From these notes it will be observed that the opinion prevails that four hours are insufficient for the complete drying of cattle foods. Some of the analysts have departed from the official method in so far as to continue the drying beyond the prescribed time, heating in some instances for six hours. Those who have conformed strictly to the requirements laid down by the association have reported results which are considerably at variance with each other. This want of uniformity in the results obtainable by the present method, together with the importance which attaches to the correct determination of moisture, seemed to the reporter to warrant special attention to this subject. A series of experiments were therefore instituted to discover, if possible, what is needed to secure a more thorough drying of feed stuffs in hydrogen.

First, it is considered of prime importance that the hydrogen used should be pure and dry. To secure this a drying battery, so to speak, was used. This consisted of a wash flask containing a solution of potassium permanganate for purifying purposes, three flasks containing concentrated sulphuric acid, and a tall calcium chlorid tower. The sulphuric acid was renewed after each experiment. It is believed that by this arrangement the gas was well dried, inasmuch as the calcium chlorid showed only slight signs of absorbed moisture, even after it had been in constant use for several weeks.

Again, the reporter was disposed to attach some importance to the velocity with which the hydrogen is allowed to flow. If it should flow too rapidly, there is danger of its not being thoroughly dry on reaching the feed stuff, and, furthermore, it would reduce too much the temperature of the latter. On the other hand the current should be fast enough to remove the vapor of water as fast as it forms, in order that the drying may be effected as expeditiously as possible.

The following tables embody the results of the investigations along this line. All the samples treated were of timothy hay, and the figures in the columns marked "hours heated" represent the per cents of moisture found. Each sample was heated for a given time (designated in hours by the number of the column in which the first per cent for that sample is given) and then heated successively for one hour at a time until the weight ceases to diminish.

Moisture determinations. (*Velocity of current of hydrogen, 40 to 50 bubbles per minute.*)

[J. T. Anderson.]

Samples.	Hours heated.								
	1.	2.	3.	4.	5.	6.	7.	8.	9.
1				11.83	11.99	12.08			
2				11.93	11.98	12.11			
3				11.84	11.92	12.05			
4				11.60	11.64	11.90			

Moisture determinations. (*Velocity of current of hydrogen, 60 to 70 bubbles per minute.*)

[J. T. Anderson.]

Samples.	Hours heated.								
	1.	2.	3.	4.	5.	6.	7.	8.	9.
1				11.73	11.76	11.81	11.83		
2				11.48	11.48	11.51			
3				11.95	11.97				
4				11.78					
5				11.99					
6				12.01					
7				11.96	12.01				
8				11.65	11.73	11.95			

Moistare determinations. (*Velocity of current of hydrogen, 90 to 100 bubbles per minute.*)

[J. T. Anderson.]

Samples.	Hours heated.								
	1.	2.	3.	4.	5.	6.	7.	8.	9.
1			11.78	11.83					
2			11.95						
3			11.88	11.99					
4			11.79	11.96					
5			11.84	11.85	11.86				
6			11.95						
7			11.93	12.07					
8			11.67	11.85					
9				12.09					
10				11.75	12.01				
11				12.11					
12				12.12					
13				11.90	12.02				
14				11.89					
15				11.93	12.08				
16				11.74	11.94				
17				12.08					
18				12.00					
19				12.13					
20				12.11					

Moisture determinations. (*Velocity of current of hydrogen, 110 to 120 bubbles per minute.*)

[J. T. Anderson.]

Samples.	Hours heated.								
	1.	2.	3.	4.	5.	6.	7.	8.	9.
1			11.65	11.65	11.74	11.85			
2			11.63	11.76					
3			11.81	11.83	11.83	11.83	11.86		
4			11.65	11.73	11.73	11.73	11.75		

The following table, which is a summary of the results contained in the four preceding tables, explains itself. Suffice it to say that the numbers in the vertical columns represent the per cent of samples by number which dried in the time specified, a sample being considered practically dry when it loses less than 10 milligrams by further heating.

Moisture determinations.

[Summary.]

Bubbles of hydrogen per minute.	Hours heated.								
	1.	2.	3.	4.	5.	6.	7.	8.	9.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
40 to 50						100			
60 to 70				62.50	75	100			
90 to 100			10	80.00	100				
110 to 120				25.00	75	100			

From this it would seem that the highest results for moisture are obtainable when the current of hydrogen flows at the rate of 90 to 100 bubbles per minute for a period of five hours. In this instance only 80 per cent of the samples dried in four hours, the time prescribed by the present official method. Some of the members of this association designate a "slow current of hydrogen" as the one employed by them. Presumably none use a slower current than 40 to 50 bubbles per minute. Even one of that velocity will not accomplish the desired purpose, it seems, under six hours. These results seem to indicate that under the most favorable conditions the limit of time prescribed by the association is too low, and should be extended at least one hour.

It should be stated that the time at the disposal of the reporter prevented any attempts being made to discover the possible effect of the more rapid currents of H on the amount of ether extract left. It is to be feared that there would be an increased tendency to carry off the volatile oils, though this was not noticeable in the coloration of the H_2SO_4, through which the gas passed.

In obedience to the instructions received from the association, the reporter has given considerable attention to the methods of ash determinations; but it must be confessed that the results which he is able to report are far from satisfactory. A number of determinations were made by each of four methods, and while there is reasonably close agreement between those made by the same method, there is considerable lack of uniformity in some instances between results obtained by different methods.

The incineration by the official method was done in a muffle furnace in a flat-bottom platinum dish, the latter being kept at a low redness until the incineration was complete. Both round-bottom and flat-bottom dishes were used, and it is believed that time is saved by the employment of the latter. The writer can not claim to have gotten a white ash in any of these determinations. Like Mr. Woll, of Wisconsin, he was forced to be content with getting a gray or grayish white ash. In our experience two grams of timothy hay under the full heat of a Bunsen flame will not burn to whiteness. The time requisite for complete incineration by this method varied from three to six hours.

The nitric acid method required less time for the incineration, and the ash was whiter than in the last method. In some cases the feed stuff was saturated directly with the fuming nitric acid, while in others it was first charred and then saturated. In all cases the excess of acid was evaporated on the water bath to prevent spirting and a consequent loss of material, and then burned in a muffle. Whether the saturation took place before or after the charring the results obtained were the same, although the process of incineration was possibly shorter when the saturation took place before charring.

The use of ammonium nitrate as an aid to combustion in ash determinations did not prove a success in our experience. It undoubtedly causes complete and rapid combustion, but the low results obtained seem to indicate that there is something wrong somewhere. In the four or five attempts we made to use it the nitrate decomposed with such vehemence as to make probable the loss of some of the material. This view of the case is strengthened by the fact that the duplicates are not concordant. We are not prepared to condemn the method, but only say that we have not yet been able to employ it successfully.

The quartz-sand method was followed in the following way: One gram each of the feed stuff and fine white quartz sand were mixed in a flat-bottom platinum dish and burned in a muffle. It required about one hour to complete the incineration. The reporter looks with considerable favor on this method. It is easy to manipulate, is rapid, and accurate in its results, at least as far as our individual experience goes. It will be observed that the United States Department of Agriculture gets results by this method which are considerably at variance with those gotten by the other methods.

The following gives an epitome of the results by the several methods of—

Ash determinations.

	Official.	HNO_3.	NH_4NO_3.	Quartz sand.
Timothy hay	5.31	5.43	4.87	5.47

There remains yet much to be done towards perfecting our methods of fat extraction, as an examination of the results reported will show. From the notes of the analysts on this subject it will be observed that in every case higher results were obtained when the extraction took place previously to drying. This is doubtless due in part to the fact that the moisture in the substance aids in the extraction and partly to the fact that the drying process affects in some way the feed stuff so as to render the extraction by ether more difficult. One thing seems to be clear, and that is that the ether used should be free from water as well as from alcohol. In our opinion the method of treatment of the ether which was adopted last year should be rigidly followed, and some efficient means for preventing the absorption of water by the ether should be adopted.

As was to be expected, there is still considerable divergence in the results on crude fiber, though it is safe to say that we are making progress on that. Your reporter ventures to express the opinion that still closer results might be reached if stronger acid and alkali solutions were employed. If $2\frac{1}{2}$ per cent solutions do not affect the fiber materially in grains it would hardly do so in the case of coarse fodders, and hence it is difficult to see what harm can come from their use in this class of goods also. Prof. Parsons used these stronger solutions in his work on the samples sent out by this reporter, and he says that his duplicates were excellent. [Prof. Jordan will doubtless make a revelation on this subject, as he has sent out timothy hay as one of his subjects for investigation.]

One word in conclusion about the necessity of titrating these solutions. The strength of the acid and alkali employed is supposed to bear some relation to the amount of carbohydrates in the substance treated. For foods and feeding stuffs low in carbohydrates we employ 1.25 per cent solutions, and for those high in carbohydrates we employ solutions of double strength. There is no line of sharp demarcation between the two classes. As to the quantity of carbohydrates, one passes by imperceptible gradations into the other. In the nature of the case the weaker solutions must be used in instances where the quantity of carbohydrates is only a little smaller than that in other substances where the solutions of double strength are called for; in other words, the relation between the strength of the solutions and the quantity of starchy matter in the one case is by no means the same as in the other. If it is not necessary, therefore, to fix a more definite and closer relation between the starchy matter and the acid and alkali, which are intended to dissolve it, it seems to me that it is needless to titrate the solutions so accurately.

RECOMMENDATIONS.

The reporter believes that great conservatism should mark the actions of this association, and no changes in the methods of analysis should be made until such changes are demonstrated to be desirable, either for the purpose of simplifying the processes or of securing more accurate results. While he thinks, therefore, that several changes might be made with profit, he has only one which he will recommend.

For the determination of hygroscopic water it is recommended that the time of drying shall be changed to five hours instead of four.

The PRESIDENT. Are there any other papers bearing on the matter? If not, are there any remarks on the recommendations?

Mr. VAN SLYKE. To get the matter before us, I move that we adopt the recommendation reported.

Mr. WOOD. I move that we substitute, as recommended, the word "five" for "four," so that the recommendation will read as follows: "For the determination of hygroscopic water, it is recommended that the time of drying shall be changed to five hours instead of four."

Adopted.

Mr. ANDERSON. The method of work as we now have it is very confusing as to nomenclature to specify what we desire. I am not prepared to suggest what it should be.

Mr. WOODS. It seems to me we ought to add after carbohydrates "high in carbohydrates other than fiber;" or, in other words, those that are rich and those not rich. I move that in both of these cases, where it says substances rich in carbohydrates and substances low in carbohydrates, that we substitute the words "other than fiber."

Mr. ANDERSON. It seems to me that we still are at sea on that subject. It would exclude what we want in this instance.

Mr. VAN SLYKE. As we seem to be at sea on the subject, I move that the matter be referred to a committee of three to look into the subject and report next year to the association.

Mr. ANDERSON. I move to amend so that they report as soon as possible.

Mr. VAN SLYKE. It seems to me we should have something to act upon during the year. Could we not decide provisionally that it shall be on substances rich and poor in fiber?

Mr. VAN SLYKE. I suggest that it might be advisable for the President to appoint two reporters who are to act upon these products next year as members of that committee.

Mr. ANDERSON. I do not know that Mr. Woods's motion received a second. I second it.

The PRESIDENT. I think the first motion was seconded. Does Mr. Van Slyke accept it as an amendment.

Mr. VAN SLYKE. I do.

The PRESIDENT. That will facilitate matters very much. It is moved and seconded, instead of having reporters upon food rich and poor in carbohydrates, that it be upon foods rich or poor in the amount of crude fiber.

Adopted.

Mr. VAN SLYKE. I move that the recommendations of the two reporters be adopted as a whole.

The PRESIDENT. It is moved that the reports as amended be adopted.

Carried.

Mr. HUSTON. I would like to have the reporter for next year requested to investigate the matter of making ether extraction without

previous drying. It is my opinion that the differences are due to actual changes in the material rather than to loss of material by volatilizing. Another thing which may perhaps claim attention is the drying apparatus used in the U. S. Internal Revenue Department.

Mr. PARSONS. I move that the reporter not only investigate the subject of water, but especially of alcohol.

Mr. HUSTON. I accept that.

Adopted.

The PRESIDENT. The next subject is the report on sugar analysis.

Mr. ROSS. I will say that quite a large portion of my paper consists of abstracts that I prepared relating to analytical methods regarding sugar, and if I can get unanimous consent of the association, I will dispense with the reading of them.

The PRESIDENT. If there is no objection, Mr. Ross is permitted to omit the abstracts. There is no objection.

REPORT ON SUGAR.

By B. B. ROSS.

In accordance with the plan heretofore generally followed in the presentation of the work of the several reporters of the association the report upon sugar analysis will consist of the following divisions:

(1) References to new analytical processes or proposed changes in existing methods, together with abstracts relating to sugar analysis, collected from various journals.

(2) Report of results of practical tests of analytical methods, both by the reporters and by chemists coöperating with him.

(3) Recommendations for the ensuing year.

Wiechman[1] has devised a method for the determination of sucrose, dextrose, and levulose in mixtures, using a combination of the optical and copper reducing methods and calculating the results algebraically from the data thus secured. A direct polarization is made with a solution containing 10 per cent of the dry substance, and reducing sugars are determined in another portion of the same solution (after proper dilution). It is claimed that, where other optically active bodies are absent, quite reliable results are secured.

O'Sullivan[2] has determined the specific rotary power of invert sugar obtained by the action of invertase upon cane sugar, and holds that results thus reached are freer from error than those obtained by inversion by means of acids.

Ost[3] has determined the specific rotary power of levulose in solutions of various degrees of concentration, and secures numbers much higher than those obtained by Jungfleisch and Grimbert, and higher than those given by Hönig and Jesser, except for more concentrated solutions. The specific rotary power of mixtures of equal weights of levulose and dextrose is found to be slightly lower than that of a solution of invert sugar of equal concentration prepared according to the Clerget-Herzfeld method of inversion, and the greater optical influence of the latter is ascribed to the presence of the strong hydrochloric acid.

Experiments made by Ost in inversion with various acids led to the conclusion that, in order to avoid destruction of invert sugar, oxalic acid (1 to 2 per cent), at a temperature of 60°—63°, is the best invertive agent.

Karcz[4] has proposed the determination of sucrose by oxidation by means of potas-

[1] School of Mines Quarterly, XIII, 3.
[2] Jour. Chem. Soc., May, 1892.
[3] Ber. d. Chem. Ges., '91. 1636.
[4] Bull. Assoc. Chim., IX, 688.

sium permanganate and sulphuric acid, carbon dioxid and water being formed. The excess of permanganate is determined by addition of standard oxalic acid and titration with standard permanganate. This method, of course, is not applicable in the presence of other bodies oxidizable by the permanganate.

Borntträger[1] notes that the maximum degree of inversion of a sucrose solution is best attained by the addition of one-tenth volume of a 38 per cent solution of hydrochloric acid and allowing the mixture to remain over night at local temperature, and recommends an invert sugar solution thus prepared for standardizing Fehling's solution.

Lacombe states,[2] as the result of a large number of observations, that different inversion constants should be used for the different temperatures at which readings of invert sugar solutions are made. For 0° the figure 142.5 is considered approximately correct, while for a temperature of 20° it is claimed that a number in excess of 144 should be employed, reaching as high as the figure 146.5 at 30°.

The same author called attention to the difference in results secured in inversion according to Clerget's method, by, in one case, plunging the flask into the water bath heated to 69° and allowing it to remain ten minutes, and, in the other case, by placing the flask in a water bath of normal temperature and allowing ten to twelve minutes for the required temperature to be attained, after which the flask is kept in the bath for ten minutes at constant temperature.

Carlier, Stift, and Lohr[3] refer to various modifications of Pellet's continuous polarization tube, with which it is claimed from 1,000 to 4,000 polarizations per day can be made by the employment of a single tube.

Schmoeger gives the conclusions derived from a series of experiments with Ost's solution, in which the advantageous points originally claimed for that solution have been practically tested.

The solution referred to consists of 23.5 grams of $CuSO_4 + 5\ H_2O$; 250 grams of potassium carbonate and 100 grams of potassium bicarbonate, dissolved in water and made up to a bulk of 1 liter.

Most of the claims are reported to be well founded, but the concentrated solution, even after some dilution, is found on boiling to give a blackish brown film on the sides and bottom of the vessel and it is sometimes the case that the precipitated suboxid is contaminated with cupric oxid.

The dilute solution is said to be free from these objections.

In sugar solutions containing calcium compounds, it is necessary to decalcify the liquid before determining reducing sugars.

Striegler, in the use of Soldaini's solution in the estimation of reducing sugars in liquids containing lime in solution, removes the lime by means of a solution of sodium oxalate and sodium bicarbonate and filters through blood carbon in order to clarify the solution and to remove reducing bodies other than sugars, these substances being, it is claimed by him, readily absorbed by this decolorizing agent.

In place of the Soldaini solution as generally prepared, he recommends the following modification:

In lieu of the 297 grams of $KHCO_3$ usually employed, 150 grams of that salt are weighed up, the remaining 147 grams being replaced by its equivalent of potassium carbonate (101.4 grams), these two salts being dissolved in 600 cc. of warm water, to which is added 100 cc. of a solution containing 6.928 grams of copper sulphate; the whole is then made up to 1 liter. The chief advantage derived from the employment of the normal carbonate is that in this way both salts can be readily dissolved in warm water, without subsequent heating and consequent loss of carbon dioxid.

Alessandri[4] proposes the estimation of glucose by means of a solution of cupro-ammonium acetate, in connection with a dilute sulphuric acid solution of standard strength.

---

[1] Scheibler's Neue Zeit., XXVII, 282.
[2] Bull Assoc. Chim., IX, 326.
[3] Chem. Zeit. Rep., 1892.
[4] Abstr. Bull. Assoc. Chim., IX, 556.

After the reduction of a portion of the copper solution by boiling for half an hour with the sugar solution under examination, the filtered liquid is treated with ammonia solution until a clear blue coloration is attained. It is then titrated with sulphuric acid until the precipitate formed just dissolves. The number of cc. thus noted is subtracted from that secured by a blank titration, and the corresponding amount of invert sugar is calculated therefrom.

Rossel[1] refers to a copper liquor in which 150 grams of glycerol per liter is substituted for Rochelle salt, the alkali employed being potassium hydrate. Borntträger[2] recommends the use of sodium sulphate instead of the carbonate for the removal of lead from sugar solutions preparatory to determination of reducing sugars, the latter salt, according to results obtained by him, tending to give results which are too high. Markiewicz[3] has given a new method for the estimation of sugar in beets, employing the Winter-Baumann extractor, using water instead of alcohol as the extractive agent, the boiling point of the water being lowered by conducting the extraction in vacuo.

Herzfeld[4] gives the result of series of experiments in the determination of water in raw sugars and sirups by means of drying both in the air bath and in vacuo. Scarcely appreciable differences are noted in the drying of raw sugars by the two processes employed, but with sirups the discrepancies in the results are larger.

The water content found by drying in the air bath at 105°–108° was in every case higher than that secured by drying in the vacuum apparatus at 90°–95°, and in the case of impure sirups, with slightly acid reaction, the results by drying in air bath are even higher than those secured in vacuo at 108°, indicating the formation of quite appreciable quantities of oxidation products in samples of this latter class, where the drying is performed in the air bath. Perfectly sound sirups were found to give, however, when dried in vacuo at 108°, higher results than were obtained in the air bath at the same temperature. Admixture with sand in drying in vacuo is found to be highly advantageous, both as regards completeness of desiccation and as to length of time consumed in the operation. For heating the vacuum drying chamber Herzfeld recommends toluene, boiling at 109°, as a substitute for a saturated salt solution, the latter being objectionable on account of the readiness with which it attacks the metal of the apparatus.

Stift,[5] in his report to the Austro-Hungarian Association of Sugar Chemists, June 20, 1892, states that in numerous tests made by him in the drying of raw sugars, even at 100°, quite perceptible decomposition has been observed, the extent of the decomposition being dependent upon the proportion of foreign substance present. Kruis[1] reports the best results as being secured by Soxhlet's drying chamber, the determination of water in massecuites and molasses being best effected by previously mixing the samples well with powdered pumice stone.

Alberti and Hempel,[6] in referring to the inaccuracies in results, as observed by them in the drying of raw sugars in air bath at 100°–110°, recommend the employment of an apparatus heated by a saturated salt solution, thus permitting the easy maintenance of a constant temperature. Babington,[7] in water determinations in sirups, employs asbestos fiber, which is saturated with a 20 per cent solution of the sample, the mass being dried in a milk tube in a steam bath for 48 hours. Pellet[8] comments upon the unreliability of the employment of density tables in the estimation of the water content of low-grade massecuites, molasses, and other sugar

[1] Abstr. Bull. Assoc. Chim., IX, 615.
[2] Scheibler's Zeit., 1892.
[3] Bull. Assoc. Chim. IX.,899.
[4] Zeit. f. Rübenzucker Ind., March, 1892.
[5] Chem. Zeit., 1892, 923.
[6] Chem. Zeit. Rep., March 19, 1892.
[7] Analyst, October, 1891.
[8] Jour. Fabr. Sucr., September 23, 1891.

products, and refers to the errors thereby occasioned in the calculation of purity coefficients.

As the result of a number of practical tests upon various factory products, he proposes the construction of tables of densities and water contents corresponding thereto for the chief sugarhouse products of different classes and grades. The data for these tables are secured by dissolving in water to a definite volume varying weights of the products employed, the weights used being reckoned upon the basis of dry substance contained. In this manner the water content as determined by taking the density and by subsequent reference to the tables alluded to will agree quite well with that obtained by actual determination.

In the estimation of ash in sugars, Paczoski[1] notes, as the result of a number of tests of Boyer's benzoic-acid method, that while results secured by this method agree quite well with each other, do not correspond with those obtained by the sulphuric-acid method, though when the ash obtained by the Boyer process is treated with sulphuric acid the agreement with the figures obtained by the usual method is nearly perfect. The oxalic method of Grobert is not found satisfactory, the combustion being slower and more difficult than with benzoic acid. Stift[2] finds the ash obtained by the oxalic-acid process to weigh the same as that obtained by simple ignition, but notes that the quantities of oxalic acid required are quite excessive.

Donath and Hattensaue[3] have tested the determination of ash by previously admixing powdered pumice stone, ignited alumina, magnesia, or copper oxid, but without satisfactory results. The incineration is found to be effected quite satisfactorily when the sample, after carbonizing, is heated, first in contact with a mixture of air and oxygen, and then in pure oxygen alone.

Alberti and Hempel[4] have proposed a method of ash determination for sugar and sugar products, which consists in thoroughly mixing the sample with a somewhat larger weight of pure quartz sand in a platinum dish, the incineration being readily effected at a moderately red heat in a muffle. The advantages claimed for this process are that by means of the thorough mixture with silica the incineration is rapid and complete, without charred residue; the silicic acid forms silicates instead of carbonates (which would ordinarily be produced), the silicic acid being substituted for the organic acids originally contained; the sulphates and alkali chlorids are not decomposed at the temperature required for incineration, and in this manner the estimation of the true inorganic constituents can be made. Sidersky[5] calls attention to the fact that Alberti and Hempel have apparently overlooked the effect the ignition with silica would have upon the nitrates contained in certain sugar products, this class of salts being completely decomposed on ignition with silica. It is admitted, however, that this difficulty could be obviated by previously making a separate determination of nitric acid. The Chemiker Zeitung[6] observes editorially that the proportion of nitrates in the German and Austrian sugars is so small that this objection is of little force with regard to the sugar products of those countries.

Donath and Eichleiter[7] report the most satisfactory results from the employment of the Alberti and Hempel method, and find that the chlorids and alkali sulphates are not decomposed by heating with quartz sand in presence of sugar, confirming in this particular the original experiments of Alberti and Hempel themselves. Weisberg[8] commends very highly the quartz-sand method for ash determinations and considers it superior to any process yet proposed.

Stift,[9] after making a series of tests of this method, reports that the claims of the

---

[1] Bull. Assoc. Chim., IX, 611.
[2] Chem. Centr., 91, 1, 445.
[3] Chem. Zeit., 1891, XV, 520.
[4] Jour. Fabr. Sucre, Aug. 26, 1891.
[5] Jour. Fabr. Sucre, Sept. 16, 1891.
[6] Abstr. Jour. Fab. Sucre, October 28.
[7] Chem. Zeit. Rep., June 4, 1892.
[8] Bull. Assoc. Chim., IX, 688.
[9] Chem. Zeit., 1892, 923,

authors in many cases do not hold good, and especially is this the case when the method is applied to low-grade products. For the purer products the results appear to be satisfactory, but for the massecuites, molasses, etc., the process, so far as it has been tested, does not seem to him to be advantageous for employment.

Courtonne[1] reports having employed ferric oxid as a substitute for silica in ash determinations, the incineration proceeding with great rapidity and results being secured practically identical with those by the quartz method.

TESTS OF ANALYTICAL METHODS.

In the latter part of the spring a circular letter was sent out to chemists in various portions of the country, requesting to secure if possible their coöperation in comparative tests of samples to be distributed by the reporter. Only eight, of about thirty chemists written to, requested that samples be sent them, and of this number only three have reported to date.

The readiness with which sugar products undergo change on standing was considered something of an obstacle in the way of securing coöperative results of value, but it was determined, nevertheless, to at least make an effort in this direction. The samples selected for distribution consisted of (No. 1) an open kettle sirup kept over from last season in a sealed vessel, and (No. 2) a low-grade centrifugal molasses found upon the market in Baton Rouge. Both samples, after remaining in the laboratory for some time, showed no perceptible tendency toward fermentation, and it was hoped that they would bear transportation without undergoing serious change in composition. Reports received from several chemists, to whom samples were sent, however, were to the effect that both samples had fermented to a greater or less degree.

The results of the analyses reported are consequently of little value as regards a comparative test of methods of analysis.

It was requested that determinations of water (solids), reducing sugars, and of sucrose, both by single and double polarization, be made, and in addition it was also suggested that in sample No. 2 a determination of reducing sugars be made in a solution clarified by lead subacetate, the excess of lead being removed from the filtered liquid by means of sodium carbonate. The results of the various determinations are given in the following tables:

Sample No. 1.

Name of analyst.	Total solids.		Sucrose.			Reducing sugars.
	Official method.	In air bath at 100–108°.	Single polarization.	Double polarization.	By inversion.	
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
E. E. Ewell, U. S. Department of Agriculture	68.76	.....	41.8	45.85	.....	16.55
T. B. Lyon, University of Nebraska	.....	.....	40.1	43.66	42.3	17.09
Maurice Bird, North Louisiana Experiment Station	.....	65.58	42.3	45.46	.....	16.33
R. E. Blouin, Louisiana State Experiment Station	.....	67.24	42.0	44.73	44.98	15.50
B. B. Ross, Louisiana State Experiment Station	68.24 *a* 66.66	67.15 *a* 66.11	41.9	44.96	44.82	15.60

a Employed sand in drying.

Total solids by density in sample No. 1 were found to be 67.7 per cent.

[1] Bull. Assoc. Chim., 1892, 806.

Sample No. 2.

Name of analyst.	Total solids.			Sucrose.			Reducing sugars.			
	Regular methods.	In air bath 105–108°.	In vacuo at 95°+.	Single polarization.	Double polarization.	By inversion.	By determination from aqueous solution.	Lead used Na_2CO_3 added before filtration.	Lead used and removed from filtrate by Na_2CO_3.	Lead used, solution acidified with acetic acid and then Na_2CO_3 added.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
E. E. Ewell, U. S. Department Agriculture	77.45	.....	.....	[1]27.8	36.42	.....	30.52	29.81	29.49	30.49
T. B. Lyon, University of Nebraska	.....	.....	.....	27.6	36.76	37.8	30.88	.....	.....	.....
Maurice Bird, North Louisiana Experiment Station	77.69	.....	.....	29.8	38.06	.....	29.59	.....	27.93	.....
R. E. Blouin, Louisiana State Experiment Station	.....	76.15	77.20	[1]27.8 [2]28.0	37.05	36.59	29.24	.....	.....	.....
B. B. Ross, Louisiana State Experiment Station	77.77	76.01	.....	[1]27.7 [2]28.1 ([3])	36.20	36.91	29.40	.....	27.02	.....

[1] Acetic acid added to clarified filtrate.

[2] No acetic acid added to clarified filtrate.

[3] By Scheibler's double dilution method 27.05 was obtained.

The differences presented by many of the results in the above table are practically almost unaccountable and irreconcilable, and it is quite difficult to draw any definite conclusions of value from the data presented. Sample No. 2 shows quite perceptibly less reducing sugars where lead and sodium carbonate have been used, but the results reported by different analysts do not agree well with each other.

Larger differences are observed in the results of double polarization than in those of single readings, but the figures by the former process are so much greater than those obtained by the latter as to emphasize the necessity of invariably resorting to the optical inversion method even where only moderately large proportions of invert sugar are present. Water determinations by drying at the temperature of boiling water show quite perceptibly lower results than are secured in the air bath at 100°—110°, and there is but little doubt that in contact with air at above 100° quite marked decomposition occurs. Sample No. 1 when mixed with sand and dried below 100° showed more than 1 per cent larger water content than when dried under same conditions without admixture of silica.

The drying of sample No. 2 was also effected in vacuo, the cylindrical drying chamber employed being heated by boiling water and a vacuum of from 24 to 26 inches being maintained. At the end of twenty-four hours drying the sample had not attained an absolutely constant weight, but indicated 59 per cent more water than was obtained by drying in contact with air.

A sample dried for fifteen hours in vacuo the chamber being heated by a saturated salt solution boiling at 107°, gave 25 per cent less water than was obtained by twenty-four hours' drying at 95°–99°, but to attain a constant weight it would have required several hours' longer drying.

The molasses gave for solids, by density, 79.6 per cent, while by actual determination the proportion of solids was only 77.2 per cent, a discrepancy which has been

often previously noted with many sugar products, and which has been practically exhibited by numerous tests made in the station laboratories in Louisiana. In order to at least approximately arrive at the water content from the Brix reading, the method proposed by Pellet and adopted by the French Sugar Chemists' Association presents many advantages which recommend its employment in sugar-house work.

An ash determination in sample No. 2 gave—

	Per cent.
By the Alberti and Hempel method	3.96
By the sulphuric-acid method	5.51
By same method with use of factor 0.9	4.96
By same method with use of factor 0.8	4.41

This shows results considerably lower for the new method, even where the smallest factor is employed in calculating results.

The quartz-sand method was also tested upon a sample of refined sugar having an ash content of 1 per cent, to which were added such quantities of potassium chlorid and sulphate as would give a theoretical ash content of 2.22 per cent. The figure obtained by actual determination was 2.14 per cent. The incineration, by this process, proceeds quite rapidly and the residue is practically free from charred particles.

In the gravimetric method for the estimation of reducing sugars it has been found advantageous to employ a modification of the electrolytic method proposed by Formanek.[1] The precipitation is accomplished as usual, a beaker being employed in the operation. The precipitated suboxid is brought upon a filter consisting of a plug of abestos placed in the neck of a funnel, the precipitate being well washed with hot water. The asbestos filter and contents are then transferred to the beaker used in the precipitation and the funnel is washed with a 4 per cent nitric-acid solution, the solution being added to the contents of the beaker until a bulk of about 200 cc. is attained. The concentric platinum cylinders are then immersed in the solution and connections are made with a battery giving a current equal to .5 to .7 oxyhydrogen gas per minute. An excellent film is thus obtained in about eight hours, and the work of manipulation requires an extremely small amount of time. The gravimetric results secured by the reporter on both samples 1 and 2 were obtained by this process, and it was noted in each case that tests failed to reveal the presence of copper in the electrolyzed liquid. The washing and drying of the films are conducted as is usual in the determinations of copper by this process.

This method, tested along with a series of volumetric estimations, gave the following results, the figures given being the amounts of invert sugar obtained by the employment of corresponding quantities of solutions of sugar products.

	Gram.
For a given volume of sugar solution the volumetric method gave	.0599
The gravimetric method gave	.0595
Another sample:	
Volumetric method	.0583
Gravimetric method	.0604
Experiment No. 3:	
Volumetric method	.0590
Gravimetric method	.0601
Experiment No. 4:	
Volumetric method	.0589
Gravimetric method	.0596

The following notes on the analytical work done were received from the coöperating chemists.

---

[1] Böh. Zeit. Zuck Ind., 1890, 14–178; Chem. Zeit. Rep., 1890, 41.

NOTES OF COÖPERATING CHEMISTS.

LABORATORY, NORTH LOUISIANA EXPERIMENT STATION.—Color polariscope employed; almost impossible to adjust tints satisfactorily on No. 2, as solution was so dark colored.

LABORATORY, NEBRASKA EXPERIMENT STATION.—Both samples had "worked" somewhat on the road. They were analyzed within two or three days after their arrival, being kept in the meantime at a temperature of about 22°. The results are stated in the order, as regards time, in which the several determinations were made.

The cupric sulphate and alkaline tartrate solutions were made up according to the formulas prescribed by the Association of Official Agricultural Chemists in 1891. In calculating results obtained gravimetrically, 0.5678 grams metallic copper is regarded as representing 1 gram reducing sugars both before and after inversion. However, all figures are given so that you can use any other means of calculating that you may see fit. The volumetric method was not used.

In determining sucrose by Clerget's method, the filtrate used for inversion was diluted to twice its volume, and the formula $R = \frac{100\,S}{142.7} - \frac{1}{2}\,T$ used. R = sucrose, S = sum of two polarizations, the second being multiplied by 2.

Hydrochloric acid of sp. gr. 1.166 was used from necessity. Five cc. were used and the solution heated for ten minutes in water already brought to and maintained at a temperature of 69° C. The polarizations were made in a 400 mm. tube provided with a water jacket, through which a stream of water was kept flowing, and the thermometer was immersed in the solution by means of a side tube. As we had considerable difficulty in filtering, the solutions were made up of only half the normal strength. Excess of lead was thrown down with sodium sulphate.

U. S. DEPARTMENT OF AGRICULTURE.—Both samples were in state of incipient fermentation when received. In the case of sample No. 1, it had proceeded so far as to necessitate the use of a little sodium chlorid in the clarification. The fermentation was stopped promptly by immersing the samples in a boiling water bath for a short time.

The clarifications were all made with basic lead acetate (with a little sodium chlorid in case of No. 1); 50 cc. of the filtrate from the lead precipitate were acidified with acetic acid and diluted to 55 cc. for the polariscopic reading. This method gains the advantage of basic lead acetate as a clarifying reagent, and avoids the error due to formation of levulosate of lead.

Total solids were determined by the method given on page 224 of Bulletin No. 31, Chemical Division, U. S. Department of Agriculture.

Reducing sugars were determined by the method (c) given on page 227 of Bulletin No. 31, Chemical Division, U. S. Department of Agriculture. Duplicate determinations were made on four solutions prepared as follows:

(1) No lead was used.

(2) Lead was added to complete precipitation, then sodium carbonate in slight excess before filtration.

(3) Lead was added to complete precipitation, the excess being removed from the filtrate by means of sodium carbonate.

(4) Lead was added to complete precipitation, and the solution made acid with acetic acid before filtration. The excess of lead was removed from the filtrate by means of sodium carbonate.

The factor 0.5698 was used for the calculation of the percentages of reducing sugars.

NOTES BY THE REPORTER.

Among the various questions bearing upon sugar analysis which have been practically tested by the reporter is that of the polarization of invert sugar solutions.

Some years since some tests were made by a committee of the Louisiana Sugar Chemists' Association with reference to the determination of the value of the constant to be employed in connection with the inversion method then in general use in that State, the process being that given by Tucker.

By this process the sugar solution is heated for fifteen minutes at 68–70° with one-tenth volume of strong hydrochloric acid. The figure obtained by these experiments was approximately 42.4, that being the observed left-handed rotation of a solution inverted in the manner described.

Similar experiments were undertaken by the reporter, aided by his assistant, Mr. R. E. Blouin, during the past season. Inversion tests conducted under the same conditions as those which were observed in making the original experiments gave results practically identical with those above referred to. In the method employed, however, a slightly higher temperature is permitted than in Clerget's original process, and the time of digestion was of five minutes longer duration.

A series of experiments were next undertaken with a view of ascertaining the rotary power of solutions inverted by acids of different strength, and with the application of heat for different periods of time. The sugar employed in the experiments was of three kinds, viz, best selected crystals of rock candy; best granulated sugars, extracted with alcohol, drained and dried thoroughly, and loaf sugar, pulverized and treated in the same manner.

The polariscopic readings were made in a room whose temperature varied from 5° to 8°, and the solutions being cooled below 0° by means of a freezing mixture, and the rise in temperature of the solutions after being placed in the observation tube was in most cases sufficiently gradual to permit of accurate observations being made.

The readings at 0° were also checked by observations made at slightly higher temperatures, due compensation being made for the decrease in rotary power occasioned by the rise in temperature. A sucrose solution obtained by dissolving a normal weight of rock-candy crystals in water, and making up to 100° was first employed, inversion being effected after addition of acid by placing in water of about 25° temperature and increasing the heat gradually until 86° was attained at the expiration of seven minutes.

The solution, when removed at this time, gave an invert reading at 0° of 41.7, indicating incomplete inversion. A portion of same solution, inverted in same manner, with the use of hydrochloric acid of 1.16 specific gravity, but maintained at 68° for four minutes, gave a reading at 0° of 45.3. When the heating was protracted for seven minutes a reading of 44.1 was secured, and at the expiration of ten minutes no further perceptible variation was noticed.

A normal sucrose solution, prepared with the use of sugar extracted with alcohol, was next treated in a similar manner, using hydrochloric acid of the same strength and heating to same temperature, though the solutions were kept in the water bath at different periods, but in every case seven minutes were consumed in heating to 68°. When heated for a period of two minutes after 68° was reached, a reading at 0° of 45.0 was obtained; at the expiration of four minutes a reading of 45.2 was made; at seven minutes a reading of 44.2 was made; at ten minutes an observation showed 44.1; at thirty minutes, 41.2, and at the end of one hour, 39.8.

Another normal sucrose solution, inverted in same manner, at expiration of three minutes, gave a reading of 45.1; at end of fifteen minutes, a reading of 43.2; after thirty minutes' digestion, an observation gave 42.0. With the use of acid of 1.20 specific gravity at end of three minutes' heating, a reading of 44.9 was noted; a result somewhat lower than that secured when 1.16 acid was employed. At the end of ten minutes' digestion the reading was 43.9, and at the end of twenty minutes a reading of 42.3 was noted. These results indicate a continued diminution of the levorotatory influence of the solutions consequent upon protracted heating in the water bath, the point of the maximum of the optical effect of the invert sugar being attained after from three to four minutes' heating at 68°.

In the inversion experiments first alluded to a slight variation in temperature of a few degrees above 68° was permitted, and as a consequence a reading of 42.4 was secured at the end of fifteen minutes, whereas when the heat was rigidly maintained at 68°, the same reading was not attained until the expiration of twenty minutes. These experiments tend to show what an important influence the degree of temperature employed and the length of time of digestion have upon the reading of solutions inverted by means of hydrochloric acid.

Quite a number of experiments were made to test the efficiency of acetic acid as an inverting agent, but it was found impossible to secure complete inversion, even when 10 per cent glacial acid was used and the flask kept immersed in boiling water for a period of forty-five minutes, thus confirming somewhat the observations of Ost.[1]

A number of tests were also made with oxalic acid as a substitute for hydrochloric acid in inversion, but it was found necessary to employ a higher temperature than 68° in order to secure the best results in the least time. Acid of about 15 per cent strength was employed and the flask containing the sugar solution, after addition of the acid, was placed in water of ordinary temperature and the heat was gradually raised until 85° was reached, after which the temperature was constantly maintained at that point. Readings were made of solutions heated at 85° for periods of from five to forty-five minutes' duration, and the figures showed scarcely any perceptible variation, being included within the limits 42.3 to 42.5.

These tests indicate that even long-continued heating with oxalic acid exercises but little effect upon the levorotatory influence of invert sugar, and the element of time is a much less important factor in the inversion process when this acid is employed.

The volume of precipitate formed where lead subacetate is employed as a clarifying agent for low-grade products exerts a much greater influence upon the sucrose percentages indicated by polariscopic tests than is generally supposed. Many authorities, however, have called attention to the errors occasioned by the bulky precipitates which remain admixed with the liquid after the solutions are made up to a definite volume. The Scheibler method of double dilution is probably the best known method of making approximate compensation for this error, but it necessitates two weighings and making up two solutions. A plan which has been tried in the laboratory of the Louisiana State University, and with quite satisfactory results, consists in making up a solution to 100 cc. in a cylinder of 200 cc. capacity. Exactly 10 cc. of the solution are filtered into a small graduate and the clear liquid is polarized in a 200 mm. tube. The funnel, together with filter and contents, is placed over the 200 cc. cylinder and a hole having been punched in the filter, the adhering precipitate is washed through, the washing being continued until the liquid in the cylinder measures 180 cc.

This last solution after mixing is passed through a filter, the filtrate being polarized in a 400 mm. tube. Subtract the last reading from the first, multiply the number thus obtained by 1.8 and subtract the product from the first reading, and the result will be the approximate per cent of sucrose. This mode of procedure involves the same principle as the method of Scheibler, and has been practically tested by the reporter, by the addition of given volumes of sand and other foreign substances to solutions containing a known weight of sugar, and comparing the actual with the calculated results. Sample No. 2, sent out by the reporter, gave for a normal weight made up to 100 cc. a reading of 27.7; but by double dilution a figure of only 27.05 was obtained. Several samples of low-grade molasses tested during the past season showed differences of more than 1 per cent by the employment of the ordinary method along with that of Scheibler.

There is also the necessity of the addition of some agent to break up the compound of lead and levulose formed when lead subacetate is added to a solution of a product containing appreciable quantities of invert sugar. Gill called attention to this

[1] Zeit. für Rübenzucker Ind., January, 1892.

source of error many years ago, but analysts, as a general rule, have failed to give heed to the necessity of making correction therefor.

A solution of invert sugar, prepared by inversion of cane sugar with dilute sulphuric acid, and neutralized with barium carbonate, was employed by the reporter in making tests of the influence of lead acetate upon the rotary power of the solution. The solution gave an original reading of —41.0 (compensated for temperature) but showed a most marked falling off as the quantities of lead acetate added were increased, at length giving a right-handed reading (compensating for dilution) in excess of the left-handed reading first observed. In every case the addition of acetic acid (the reagent usually employed for this purpose) causes the restoration of the rotary power of the solution and its addition, or that of some substitute, is especially essential where double polarization is resorted to. Prof. Crawley, of the sugar experiment station of Louisiana, obtained results quite similar to those above reported.

By reference to the table of results it will be noticed that sample No. 2 gave a polarization of 28.0 without addition of acetic acid, while with this acid a figure of 27.7 was obtained.

Mr. Edson, of the U. S. Department of Agriculture, some years since, referred in a pamphlet to the discrepancies noticed in the determination of reducing sugars in cane juices by estimation from simple aqueous solutions, and from solutions to which lead subacetate had been added, the excess of lead being removed by precipitation. In consequence of the precipitation of reducing bodies by the basic lead acetate, Mr. Edson has proposed the use of the neutral acetate in preserving samples, this latter salt, it is claimed, being free from the objections urged against the basic compound. Prof. Crawley called attention during the past spring to some results secured in the estimation of reducing sugars in molasses to which lead subacetate had been added, there being quite a considerable difference in the results secured where sodium carbonate was added, as a precipitant to the original solution and where it was added to the filtered liquid. To test this question further the following experiments were made by him.

Forty grams of a centrifugal molasses were dissolved in water and made up to a volume of 1 liter, and different portions were treated as follows and gave results as herewith stated.

(1) 50 cc. made up to 100 cc.; 10 cc. Fehling's = 13.5 cc. molasses solution = 18.52 per cent reducing sugars.

(2) 50 cc. + 3 cc. lead subacetate, a gram of Na_2CO_3 added, the solution diluted to 100 cc. and filtered; 10 cc. Fehling's = 13.6 cc. solution = 18.38 per cent reducing sugars.

(3) 50 cc. + 3 cc. acetate of lead + acetic acid to neutralize, diluted to 100 cc. filtered. 10 cc. Fehling's = 13.5 cc. solution = 18.52 per cent.

(4) 50 cc. + 3 cc. subacetate lead made to 100 cc., filtered, added sodium carbonate, filtered. 10 cc. Fehling's = 15.0 cc. solution = 16.67 per cent.

A sample of sorghum molasses, treated as above, gave the following results:

(1) Treated as 1 above: 10 cc. Fehling's = 13.1 cc. solution = 19.08 per cent.

(2) Treated as 2 above: 10 cc. Fehling's = 13.4 cc. solution = 18.66 per cent.

(4) Treated as 4 above: 10 cc. Fehling's = 14.4 cc. solution = 17.36 per cent.

Molasses from sugar house of the Experiment Station:

(1) As 1 above: 10 cc. Fehling's = 14.8 cc. solution = 16.89 per cent.

(2) As 2 above: 10 cc. Fehling's = 14.8 cc. solution = 16.89 per cent.

(4) As 4 above: 10 cc. Fehling's = 15.8 cc. solution = 15.82 per cent.

At the same time, the reporter made a series of tests in the laboratory of the State university in order to arrive at an understanding of the causes of the divergent results above alluded to. Five samples of low-grade molasses were employed in the tests referred to.

In the following table, Column A gives the number of cc. of an aqueous solution of the molasses required to reduce the copper in 10 cc. of Fehling's solution. Column B gives the number of cc. of an alcoholic solution required to reduce the same amount of copper solution. Column C gives the figures for solutions clarified by lead acetate, the lead being precipitated from the filtered solution by means of sodium carbonate, and the liquid being again filtered. Column D gives the results obtained from solutions where sodium carbonate was added directly to the solution containing lead, without previous filtration of the liquid.

Column E gives the results for an aqueous solution decolorized by bone black.

Sample.	A.	B.	C.	D.	E.
No. 1	22.0	22.0	26.6	23.4	.....
No. 2	24.0	25.0	29.2	25.4	.....
No. 3	27.3	28.0	32.6	29.6	.....
No. 4	29.0	29.2	32.6	29.5	29.5
No. 5	29.4	29.6	35.7	30.8	30.9

By reference to the figures above it will be seen that in all cases the per cent of reducing sugars is very much smaller where the solution, after the addition of subacetate of lead, is filtered before the addition of sodium carbonate, and that where sodium carbonate is added before filtering the results are almost identical with those obtained where nothing was added to the water solution. This shows that reducing sugars are precipitated by lead subacetate and filtering removes them. If, however, sodium carbonate is added before filtration to precipitate excess of lead, the greater quantity of these reducing bodies are probably set free and pass through the filter, and further experiments tended to confirm this opinion.

The fact, too, that the solutions to which lead acetate and sodium carbonate were added, with an intervening filtration, exerted a less reducing effect than did solutions made up according to the ordinary methods, showed plainly that some of the bodies, removed by the lead, possessed quite an appreciable reducing power.

It was at first supposed that some of these bodies were, perhaps, precipitable by alcohol, as well as lead subacetate, but a number of determinations of reducing sugars from alcoholic solutions showed but slight variations from results secured under normal conditions.

As the addition of sodium carbonate along with lead subacetate gave a solution more highly colored, and at the same time of a greater reducing power, than where filtration was resorted to before the precipitation of excess of lead, the indications seemed to point to the reduction of the copper solution by some of the coloring matters present. Some of the samples were therefore made up with water as usual, the very dilute solutions decolorized by boneblack and tests of the reducing effects of the clear liquid upon the copper solution were made, with the result that there was noted but little variation from figures obtained by the use of the ordinary methods. This, of course, indicated that the reducing bodies, other than sugars present, were not precipitable by alcohol, nor were they included in the coloring matters removed from the molasses.

In order to determine what reducing body or bodies other than sugars were present, a larger proportion of molasses was taken and the aqueous solution was filtered through several layers of boneblack, a practically colorless filtrate being secured. An excess of lead acetate solution was added, and the solution, after being well agitated, was allowed to stand, and the precipitate, after settling, was removed by filtration, and, after being well washed, was transferred to a beaker, suspended in water, and a stream of sulphuretted hydrogen was passed through the liquid, in order to precipitate the lead as sulphid. After filtration the liquid was evaporated to expel sulphuretted hydrogen, and the concentrated solution showed quite an acid

reaction. A portion of the solution, on being subjected to polarization, gave a left-handed reading, and a solution obtained in a similar manner from another sample of molasses showed a like optical influence. Another portion of solution, on being tested for its reducing effect upon copper solution, was found to precipitate the copper in the proportion of 41.2 cc. of the Fehling solution to an equivalent of 10 grams of molasses.

In order to test acidity of the solution, a portion was titrated with 1.40 normal sodium hydrate solution, using phenolphthalein as an indicator, and it was found that 36.6 cc. of the alkali were required to neutralize the acid obtained from 10 grams of molasses, indicating the presence of an equivalent of .252 gram of malic acid. It was found impossible to precipitate the acid present with calcium chlorid in the concentrated aqueous solution even on boiling, but on addition of alcohol the precipitation was complete, being redissolved, however, with readiness on decanting the alcohol and adding water. It was found upon examination that the concentrated solution reduced potassium dichromate solution in presence of sulphuric acid on application of heat, a faint greenish tinge being produced and a characteristic apple odor being at the same time noticeable.

The sample of molasses sent out by the reporter was also treated in the manner above described, and there was found of reducing bodies, precipitable by lead, an amount equivalent to .0086 grams of invert sugar per each gram of molasses (equivalent to .86 per cent). The results of these experiments undoubtedly indicate the presence in molasses of quite appreciable quantities of reducing substances precipitable by lead subacetate.

It was observed that the precipitate thrown down from the copper solution by the reducing bodies referred to did not, even at the end of the reaction, possess the bright red color ordinarily produced in the determination of reducing sugars.

Tests made by the reporter with additions of varying proportions of lead subacetate to invert sugar solutions of different degrees of concentration failed to show any perceptible precipitation of invert sugar by the lead.

RECOMMENDATIONS.

The reporter makes the following recommendations for the ensuing year:

On page 225, Bulletin 31, after third line, insert "in case of products in which incipient fermentation is indicated, the drying is to be preferably conducted in vacuo."

On same page, strike out the last paragraph under the heading of "density and total solids."

Under the head of the estimation of reducing sugars, it is recommended that the revised methods proposed by Dr. Wiley be substituted for the present official methods.

It is also recommended that in the electrolytic estimation of the reduced copper, the modified method of Formanek be inserted for provisional use. That under the section relating to determination of sucrose there be inserted detailed directions for preparation of solutions for polarization. Where double polarization is employed, it is recommended that acetic acid be added to the solution in a proportion sufficient to break up the compound of lead with levulose previous to making the direct reading.

It is further recommended that a method for the preparation of basic lead acetate and alumina cream be inserted.

Also that Clerget's original method for inversion be included among the association methods.

That the German official process for inversion, described in Bulletin 28 as among the elective methods (pp. 217–228), be also inserted under the head of optical methods.

Under the head of ash determination, section (*e*), it is suggested that a definite factor be employed in the calculation of results.

Among the methods to be tested for the ensuing year the reporter recommends that

the Pellet process for estimation of water in molasses, massecuites etc. (described in one of the abstracts) be subjected to practical trial.

That the Alberti and Hempel methods for ash determinations be also tested by the reporter for next year.

Abstracts Relating to Sugar.

A Method for the Estimation of Sucrose, Dextrose, and Levulose in Mixtures. F. G. Wiechmann.

The substance under examination is made up in aqueous solution on the basis of 10 grams of dry substance to 100 grams of solution. A portion of the solution is polarized, the result being recorded in circular degrees. Reducing sugars are determined in the same solution, considerably diluted, both before and after inversion, the amount of sucrose thus found being calculated in the usual manner. The algebraic calculation of results is performed as follows:

Let:

a = amount of sucrose present.
b = amount of total reducing sugars present.
x = amount of dextrose present.
y = amount of levulose present.
s = the specific rotatory power of sucrose, divided by 100.
d = the specific rotatory power of dextrose, divided by 100.
l = the specific rotatory power of levulose, divided by 100.
p = polarization observed, expressed in circular degrees.

Then

$$(as + xd) - yl = p.$$
$$(as + xd) = p + yl.$$
$$xd = p + yl - as.$$
$$x = \frac{p + yl - as.}{d}$$

Substituting this value of x in the equation

$$x + y = b$$

there results:

$$\frac{p + yl - as}{d} + y = b.$$
$$p + yl - as + yd = bd.$$
$$yl + yd = bd - p + as.$$
$$y\,(l + d) = bd - p + as.$$
$$y = \frac{bd - p + as}{l + d.}$$

As y represents the amount of levulose, y deducted from b will give at once the amount of dextrose. Or, if preferred, the value of x can also easily be calculated independently.

The results of a number of practical tests of the method are given, and it is claimed that the estimations by this process are quite reliable where optically active bodies other than sugars are absent.

Determination of Sugar in the Beet by Aqueous Extraction. Markiewicz.[1]

The determination is effected by the use of the Winter-Baumann extractor, employing water in place of alcohol. As the boiling point of the aqueous extract is higher than that of the alcoholic the author has made use of the vacuum to facilitate the operation, the apparatus being connected either with an air pump or with the

[1] Bull. Assoc. Chim., IX, 899.

condenser of the quadruple effect of the factory. The comparative tests of the aqueous and alcoholic extraction processes give results whose mean is about 0.1 per cent in favor of the aqueous method.

Determination of Sucrose by Oxidation. Karez.[1]

The estimation is effected by oxidation by means of H_2SO_4 and $KMnO_4$. The decomposition is represented by the equation

$$C_{12}H_{22}O_{11} + 24\,O = 12\,CO_2 + H_2O.$$

In a flask of 350 cc. capacity dissolve .1271 grams of sugar in distilled water. Add exactly 50 cc. of a standard solution of permanganate, containing in 1 cc. .011734 grams of $KMnO_4$. Shake well and then add 20 cc. of concentrated H_2SO_4. After two minutes dilute with 100 cc. of water, add 50 cc. $\frac{N}{10}$ oxalic acid, and heat to boiling.

The liquid is then titrated with permanganate solution. The method is not applicable to raw sugars containing other substances oxidizable by the permanganate.

The Specific Rotary Power of Cane Sugar in Dilute Solutions. R. Nasini and V. Villavecchia.[2]

The results previously obtained with regard to this subject are somewhat conflicting.

The authors, working with pure cane sugar and using a Landolt-Lippich polaristrobometer, have obtained at 20° for a solution containing—

1.2526 per cent sugar $(a)_D = 66.604$ (observed) against $[a]_D = 66.783$ (calculated).
For 1.2048 per cent sugar $(a)_D = 66.855$ (observed) against $[a]_D = 66.785$ (calculated).
For 0.8243 per cent sugar $(a)_D = 67.250$ (observed) against $[a]_D = 67.213$ (calculated).
For 0.5982 per cent sugar $(a)_D = 67.562$ (observed) against $[a]_D = 67.715$ (calculated).
For 0.3351 per cent sugar $(a)_D = 68.241$ (observed) against $[a]_D = 68.539$ (calculated).

The calculated rotary powers were derived from the equation

$$(a)_D = 69.962 - 4.8696\,p + 1.86145\,p^2.$$

The results do not agree with those of Tollens and Pribram.

The Inversion of Cane Sugar by Means of Brewers' Yeast.

C. O'Sullivan and F. W. Tompson[3] have proposed to employ brewers' yeast in the estimation of cane sugar by inversion, the mode of procedure and of calculation of results being somewhat similar to the process with invertase.

Fifty cc. of the solution of the sugar to be examined are placed in a beaker and raised to 55° by means of a water bath. Pressed brewers' yeast should now be added in the proportion of about one-tenth of the weight of the sugar present, and the solution should be maintained at a constant temperature for four hours. The solution is then cooled, filtered, made up to 100 cc., and both polariscopic and cupric reducing tests are made. In calculating results the factors employed are derived from results of parallel experiments with pure cane sugar.

Optical and reducing tests should be made before the inversion of the sample, and the solution should be approximately neutral and free from any enzyme. The results are reported to correspond closely with those obtained by the use of invertase.

Polarization of Sugar Solutions Inverted by HCl. Borntträger.[4]

The rotary power of neutralized solutions, when diluted, is higher than that of acid solutions for same degree of dilution.

Concordant results are secured by neutralizing after inversion with NaHO, KHO, Na_2CO_3, CaO, BaO, MgO, then acidifying feebly with acetic acid, diluting to 100 cc., and polarizing after several hours; otherwise the errors may be considerable. In one case —16.4 was obtained for an acid solution, while for a neutral solution of same concentration —17.0 was secured.

---

[1] Strohmer's Zeitschrift, xx-698. Bull. Assoc. Chim., 92-689.
[2] Gazzetta, 22, I, 97. Jour. Chem. Soc. Abstr., 1892, 891.
[3] Trans. Chem. Soc., 1891.46. Bull. Soc. Chim., Paris, April, 1892.
[4] Boh. Zeit. Zucker-Ind., 1891, 187. Bull. Assoc. Chim., 1892, 559.

The Specific Rotary and Cupric Reducing Power of Invert Sugar and of Dextrose obtained from Cane Sugar by means of Invertase. J. O'Sullivan.[1]

The author remarks that the numbers heretofore published for the specific rotary and cupric reducing power of invert sugar and of dextrose prepared from invert sugar have been determined from the products obtained from a solution of cane sugar by heating it with an acid.

After referring in detail to the numbers heretofore given by various authorities for the specific rotary power of levulose and invert sugar, the results of a series of experiments upon the inversion of cane sugar by means of invertase are given.

Pure crystallized sugar candy was employed in the inversion tests, a solution in water being prepared which contained 11.261 grams in 100 cc. of the solution.

About one-fourth of this solution was digested with .03 grams of invertase for seventeen hours at a temperature of 14–16°. The specific rotary power of the invert sugar in this solution was found to be $[a]j = -24.1$ at 15.5°.

The cupric reducing power of the invert sugar, as determined by O'Sullivan's modified gravimetric method, was found to be K = 101.

Experiments with cane sugar dissolved in water and crystallized from ethyl alcohol were also made, the inversion being conducted as in the manner before described.

The specific rotary power of the invert sugar solution thus obtained was (C = 11.4) $[a]j = -24.8$ and the cupric reducing power K = 99.

With a solution of greater density prepared from the large cane-sugar crystals, the specific rotary power of the invert sugar was (with a concentration of 14.07) $[a]j = -24.5$ and the cupric reducing power K = 99.3.

Dextrose was also prepared from a cane-sugar solution inverted by invertase, the crystals obtained from the evaporated syrup, being washed with alcohol, purified by recrystallization from water and methyl alcohol, and finally dried in vacuo over H_2SO_4.

At 15.15° the numbers representing the specific rotary power, as shown by two experiments, were $[a]j = 57.1$ and $[a]j = 57.2$, respectively.

The cupric reducing power as indicated by the means of two tests was K = 100.

As the specific rotary power of invert sugar obtained by means of invertase where levulose is not acted on, is $[a]j = -24.5$, and that of the dextrose prepared from it is $[a]j = 57$, the apparent specific rotary power of levulose calculated from these numbers must be $-24.5 \times 2 + 57 = [a]j = -106$, $[a]D = -93.8$, invert sugar being composed of equal portions of levulose and dextrose.

The Rotary Power of Levulose and of Invert Sugar. H. Ost.[2]

From previous experiments made by the author, he was led to believe that crystallized levulose possessed a greater rotary power than that given by Hönig and Jesser (Zeit. für Rübenzucker-Ind., 1888—1027). Jungfleisch and Grimbert (Compt. Rend., 107—390) obtained other numbers, as did also Wohle[3], so that many chemists claim that there are different levuloses.

The levulose used was prepared from inulin and purified by crystallizing three to four times from absolute alcohol, the product being then apparently identical with that of Hönig and Schubert.

It crystallized in compact prisms having excrescences upon the surfaces, which dried over H_2SO_4 are always water free.

On the contrary, the crystallized levulose of Jungfleisch and Grimbert contained $2\frac{1}{2}$ per cent of water.

The preparation used by the author was not at all hygroscopic, 1.52 grams of it, dried over sulphuric acid, not gaining so much as 1 mgm. in weight on exposure to air.

Heated in a stream of dry hydrogen, it melted and gradually lost weight 0.75 per cent in ten hours, without reaching a constant weight.

---

[1] Journal Chem. Society, Trans. 1892, 408.

[2] Ber, Chem. Ges., 1891—1636. Zeit. des Ver. f. die Rübenzucker-Ind., 1892–47.

[3] Ber. Ch. Ges., xxiii—2099.

The author takes issue with the statements of Jungfleisch and Grimbert, that an aqueous solution of levulose undergoes slight decomposition when heated to 50° and that a quite considerable decrease in rotary power takes place at 100°.

He remarks that, even in the evaporation of aqueous and alcoholic solutions of pure levulose, there is no danger of a destruction of its rotary power. Five different preparations were employed by the author, being obtained from the crystallization of different portions of pure alcoholic solutions of levulose.

In determining the rotary power of the levulose all weighings were reduced to a vacuum and all polariscopic readings were made at 20° each reading reported being the mean of several individual observations. Fifteen solutions, containing from 1.01 to 30.11 per cent levulose, were prepared, giving specific rotary powers of $[(a)\ \frac{20}{D}] = -90.64$ to -95.39. For the solutions containing from 3 to 30 per cent levulose, there were secured results from which the following formula was deduced:

$$(a)\ \frac{20}{D} = -(91.90 + 0.111\ p)$$

The author has plotted the values obtained by him, together with those secured by Hönig and Jesser, and those of Jungfleisch and Grimbert. Ost's curve is higher than that of Hönig and Jesser for solutions containing less than 25 per cent levulose and lower for solutions of greater concentration, while it is about 3 per cent higher than that of Jungfleisch and Grimbert throughout. This latter difference is ascribed to the possible presence of optically inactive bodies in the levulose employed by the last-mentioned authorities.

As with dextrose (as observed by Tollens), the specific rotary power undergoes a considerable decrease with the dilution of the solution.

The author next prepared solutions containing mixtures of equal weights of levulose and dextrose for the purpose of comparing the results practically obtained by polarization with the calculated results.

The dextrose used was after drying at 100°, crystallized from absolute methyl alcohol and dried over H_2SO_4.

In making the calculations, the author's numbers for levulose and those of Tollens for dextrose were employed.

The results secured practically are throughout slightly higher than the calculated values, and this difference is assumed to be possibly due to the fact that Tollens dried his dextrose at 60–70°, whereby it might have suffered a slight change.

With dextrose, dried over H_2SO_4, the following numbers were obtained—with p=10; $(a)\ \frac{20}{D} = 52.54$, as against 52.74 by Tollens; with p=2: $(a)\ \frac{20}{D} = 52.15$.

It is observed that inverted cane-sugar solutions possess a different rotary power according to the manner of inversion.

According to the Clerget-Herzfeld method of inversion, the mean of three determinations gave for the specific rotary power — $(a)\ \frac{20}{D} = -20.71$ (the number obtained by Herzfeld being —20.63), this figure being considerably above that for a pure invert sugar of equal concentration, $(a)\ \frac{20}{D} = -20.28$.

This the author ascribes to the previously observed tendency of hydrochloric acid to increase the rotary power of invert sugar solutions.

Gubbe has avoided this incidental increase in rotary influence by the employment of oxalic acid as an inverting agent, obtaining a number 0.2° below that given above.

Jungfleisch and Grimbert have claimed, however, that oxalic acid increases the rotary power of invert sugar; that acetic acid is without influence in this respect and that it completely inverts cane sugar at 100°.

To test the reliability of these claims, experiments were made with 13 per cent solu-

tions of a mixture of equal quantities of levulose and dextrose, heating the same with both oxalic and acetic acid at different temperatures.

Heated with 1 per cent oxalic acid for three hours, at 50–53°, the optical activity of the invert sugar is not perceptibly decreased, while with a heating of one-half hour at 100° with 5 per cent acetic acid, a marked diminution in rotary power is observed (from 20.3 to 19.77).

A solution of levulose when heated with one-fourth per cent oxalic acid fifteen minutes at 100° showed a reduction of rotary power from —93.34 to —92.02, while 1 per cent hydrochloric acid, under same conditions, gave the figure —92.19.

Experiments upon the inversion of sucrose solutions showed that the inversion was best effected by heating with 2 per cent oxalic acid for 1½ hours at 60°, or with 1 per cent oxalic acid for 4 hours at 60° to 63°. With 5 per cent acetic acid inversion was incomplete, even after heating 1 hour at 100°, and it is therefore considered unreliable as an inverting agent.

It is noted, in conclusion, that an invert-sugar solution prepared according to Soxhlet, with use of 1 per cent hydrochloric acid at 100°, is not entirely identical with a strong sugar solution inverted according to the Clerget-Herzfeld method.

Experiments with invert-sugar solutions prepared after Soxhlet's directions shows that its reducing effect upon copper-potassium carbonate solutions is practically identical with that of a mixture of pure levulose and dextrose. It is therefore to be recommended for employment in the standardization of copper solutions.

Note by Tollens on the Preceding Paper.[1]

The attention of Ost is called to the fact that he has overlooked the report of the investigations by Parcus and Tollens upon the specific rotary power of levulose, they having obtained for a 10 per cent solution the numbers $(a)\ D = -92$ to -92.5 (one time — 93.03).

Regarding Ost's statement that the numbers obtained by Tollens for dextrose were probably 0.1 to 0.2 high, by reason of the dextrose having been dried at 60° to 70°, it is observed that it is quite likely that Ost's preparation, dried over sulphuric acid, still contained about 2 per cent water.

The Influence of Bone-black upon Sugar Solutions.

Martina[2] notes, as the result of a number of tests of clarifying agents, that in the employment of bone-black as a decolorizer the following effects were observed:

(1) That the amount of decrease in the polarization of a solution is dependent upon the amount of bone-black employed, a definite amount of sugar being absorbed by a given weight of the animal charcoal.

(2) That the revivified black possesses the peculiar property of absorbing and retaining less sugar than the new bone black.

(3) That the diminution in polarization produced by a constant quantity of bone black is the same, whatever may be the richness of the sugar solution.

Clerget's Inversion Process, as Recommended by the "Assoc. des Chimistes."[3]

Fifty cc. of the defecated and filtered liquid are placed in a flask graduated to 50 to 55 cc. and 5 cc. pure fuming HCl are added. Then agitate contents of flask well and place in a water bath with double bottom.

The temperature is raised by means of an alcohol lamp or Bunsen burner to 68° the flame being so regulated that from ten to twelve minutes are required in reaching this temperature. At the end of twelve minutes the flask is removed from the water bath and is cooled to local temperature by immersion in a vessel of cold water.

After the cooling is accomplished it is filtered (if necessary) without alteration of volume.

It is then polarized in a 22 cm. tube and results are calculated according to Cler-

[1] D. Chem. Ges. Ber., 1891, 2,000; Zeit. F. Rübenzucker-Ind., Jan., 1892.

[2] Stazioni sperimentale agrarie Italiane, 91; Bull. Assoc. Chim, 1892, 616.

[3] Jour. des Fabr. de Sucre—XXXII, 1891, No. 35.

get's original formula. If the liquid is colored, about 1 gram of dry pulverized bone black is added previous to filtration.

A Crystalline Magma of Invert Sugar.

Wiechmann,[1] in testing the influence of light upon invert-sugar solutions, found that after three months' standing an 85 per cent solution yielded a snow-white crystalline magma. The magma on examination showed 97.06 per cent invert sugar and 2.8 per cent cane sugar, reckoned on the basis of dry substance present. The specific rotary power, compensating for presence of sucrose, was found to be for a 10 per cent solution $[a]\ D^{20} = -20.027$ as against -20.0181, as given for pure invert sugar by Gubbe.

Detection of Small Quantities of Sugar. Müller and Ohlmer.[2]

Into a graduated cylinder 2 cm. in diameter and 10 cm. high, place 2 cc. of the sugar solution to be tested (condensings, etc.); next, by means of a dropping glass, 5 drops of a pure 20 per cent alcoholic solution of α-napthol are added and then 10 cc. of pure concentrated sulphuric acid, absolutely free from nitric acid, the mixture being well shaken; after some minutes the color produced reaches its greatest intensity.

With 0.1 per cent of sugar content there immediately arises a very intense violet color; with 0.01 per cent sugar a wine-red color is produced; with 0.001 per cent, after one-quarter to one-half a minute, and with 0.0005 per cent after several minutes a bright rose color appears; with 0.00001 per cent no reaction was perceptible. Calcium salts, chlorids, ammonia, organic substances (excluding the carbohydrates) do not disturb the reaction, but traces of nitric acid in dilute solutions interfere with it.

Sugar Tests with α-Naphtol.[3]

Thiele, enlarging upon Müller's last statement with reference to this subject, notes that a test for 0.00001 per cent sugar is not to be obtained if one proceeds exactly according to Molisch but if to 5 cc. of the liquid under examination only a drop of 4 per cent alcoholic α-napthol solution is added, followed by 1 to 2 cc. of concentrated H_2SO_4, the mixture being shaken carefully, after some minutes there appears a clear, rosy coloring, which with standing becomes dark.

The author concludes that, quantitatively, this reaction is not useful, the differences of color being too slight.

Solubility of Sugar in Water. Herzfeld.[4]

The results of experiments of Michel and Krafft, Anthon, Marschall, Scheibler, Flourens, and others are referred to, and the author presents data secured by himself with reference to the solubility of sugar in water at various temperatures.

An apparatus of special construction, permitting of the most accurate regulation of temperature, was employed in making the solubility tests, observations being made at temperatures varying from 5.2° C. to 99.45° C. At 5.2° C. the percentage solubility was 65.17; at 19.15° it was 66.65; at 28.8°, 68.31; at 49.53°, 72.23; at 59.4°, 74.33; at 99.45°, 82.76. From the data thus obtained the following equation of the curve of solubility was deduced:

---

[1] School of Mines Quarterly; Louisiana Planter, July 9, 1892.

[2] D. Zucker-Ind., 1892, 17–419; Chem. Zeit. Rep., XVI, 1892, 126.

[3] D. Zucker-Ind., 1892, 17–456; Chem. Zeit. Rep., 1892, XVI, 126.

[4] Zeitschrift f. des Vereins f. die Rübenzucker-Ind.

$$y = 64.1835 + 0.13477\ x + 0.0005307\ x^2$$

in which x = number of degrees Centigrade and y = per cent sugar in solution at $x°$ C.

From this the following table was prepared:

Temperature, C.	Percentage of sugar.	Temperature, C.	Percentage of sugar.	Temperature, C.	Percentage of sugar.
°		°		°	
0	64.18	35	69.55	70	76.22
5	64.87	40	70.42	75	77.27
10	65.58	45	71.32	80	78.36
15	66.33	50	72.25	85	79.46
20	67.09	55	73.20	90	80.61
25	67.89	60	74.18	95	81.77
30	68.70	65	75.18	100	82.97

The results of Herzfeld and Flourens exhibit the greatest differences between the temperatures of 5° and 50°, the figures of the former being from 3 per cent to 75 per cent higher.

In testing the influence of the presence of salts upon the solubility of sugar in water it was found that, in general, small quantities of salts decreased the solubility, while large quantities, either of single salts or of mixtures of salts, produced contrary effects. The salts employed in mixtures included the nitrate, tartrate, malate, citrate, acetate, and lactate of potassium and the chlorid of sodium.

The author found that when small quantities of such salts as potassium chlorid and acetate were added to a saturated sugar solution, a small proportion of the sugar was precipitated, but was again brought into solution upon the addition of an excess of those salts.

Small quantities of colloid bodies, such as albumen, pectin, dextran, dextrin, etc., are found to decrease to a slight extent the solubility of sugar in water.

The Influence of Boiling upon Sugar Solutions. By J. Weisberg.[1]

The results of experiments of Soubeyran, Fensky, Morin, and others, it is claimed, show that sugar solutions on prolonged boiling exhibit quite a marked decrease in rotary power.

The author, in order to test the correctness of the conclusions drawn from their experiments, made a series of tests upon pure sucrose solutions, the liquids being subjected to boiling for periods of from 2 to 12 hours duration. The solutions were boiled in a retort supplied with reflux condenser, so that a constant volume of liquid could be maintained, while a vigorous ebullition was continually secured by the direct application of the flame of a burner to the gauge on which the retort rested.

The original solution and the corresponding boiled solutions were polarized under like conditions.

		Polarization in 400-mm tube, mean of several readings.
Experiment 1.	(*a*) Original solution	7.40
	(*b*) After 3 hours' boiling	7.40
	(*c*) After 5¼ hours' boiling	7.40
Experiment 2.	(*a*) Original solution	45.66
	(*b*) After 3 hours' boiling	45.53
	(*c*) After 12 hours' boiling	45.18
Experiment 3.	(*a*) Original solution	44.37
	(*b*) After 2 hours' boiling	44.34
	(*c*) After 8 hours' boiling	43.66

[1] Bull. Assoc. Chim., IX, 11, 862; Scheibler's Neue Zeit., XXVIII, 23-279.

The solutions which had been boiled longest showed quite a perceptible darkening in color and had a slight reducing effect upon Fehling's solution.

The falling off in the polarization of the sugar solutions was somewhat more marked when traces of the following salts were present in the solutions subjected to boiling, viz, oxalate, nitrate, and sulphate of potassium, acetate and phosphate of sodium.

The author concludes that sugar solutions can be maintained at a temperature of from 100° to 105° for quite a long time without undergoing an appreciable change, though with still more protracted boiling the polarization decreases and a reducing influence is exerted upon Fehling's solution.

Soubeyran's results are considered entirely unreliable, the author only attaining results of a like character by boiling a solution with a drop of acetic acid.

The influence of lime upon boiling sugar solutions was tested by mixing milk of lime with a pure sucrose solution and boiling the filtered liquid in the retort supplied with reflux condenser. After three hours' boiling but little change had taken place in the rotary influence of the solution, but after an interval of eight hours there was a decrease in polarization of 0.49 per cent.

Formula for Estimation of Sucrose and Raffinose by Inversion. Herles.[1]

The variation in polarization (due to temperature) of a normal sucrose solution after inversion is, according to Clerget, indicated by this formula, in which s is the reading at 0° of an inverted sucrose solution that read 1° on the sugar scale before inversion: Reading at t° of an inverted sugar solution, that read 1° on the sugar scale before inversion $= s + 0.005\ t$.

According to Herzfeld the variation for raffinose is $0.18\ t$ for 100° (of sugar scale), and according to Creydt, $= 0.15\ t$. However, without great error, $0.2\ t$ may be employed.

If r is the polarization of inverted raffinose for 1° of direct polarization at 0° C., at t° it is equal to

$$r + .002\ t$$

Let S = sucrose, R = raffinose, P = polarization (direct), I = polarization after inversion, then—

(1) $$P = S + 1.85\ R.$$

(2) $$I = (s + .005\ t)\ S + 1.85\ R\ (r + .002\ t)$$

from which are obtained:

$$S = \frac{(r + .002\ t)\ P - I}{(r - s) - .003\ t}$$

$$R = \frac{P - S}{1.85}$$

These formulæ are independent of the value of the inversion constants employed. According to Herzfeld the inversion constant for sucrose is, at 20°, −32.66. According to Herzfeld and Tollens, an original polarization of 100° for raffinose corresponds to 51.29 after inversions at 20° C. Taking account of temperature,

$$r = .4724$$

Introducing these values in above formulæ,

$$S = \frac{(.4724 + .002\ t)\ P - I}{.899 - .003\ t}$$

If $t = 20°$,

$$S = \frac{.5124\ P - I}{.839}$$

It must be remembered that s is a negative quantity, and that I may be positive or negative according to the ratio of sucrose to raffinose.

[1] Nevole's Zeitschrift, 1891, 8, 528, Bull. Ass. Chimistes, 1892, 691.

The Inversion and Estimation of Raffinose. Koydl.[1]

The method of Herzfeld is employed, but the solution of one-half normal weight of sugar in water is made up to 250 cc. instead of 100 cc.

Herzfeld's constant (—32.66) has been fully confirmed.

The author has prepared a table of inversion constants, whose values are dependent upon the proportion of ash constituents present and the quotient of purity of the product. The data from which the table was constructed were the results of a series of experiments upon solutions of sugar containing varying proportions of sodium chlorid and acetate and potassium nitrate.

It is recommended that the regular raffinose formula be not employed in sugar tests unless the presence of raffinose is positively known, Clerget's formula being preferable in absence of that body.

Raffinose Determinations. Weisberg[2].

The author confirms the statements of Koydl and, in addition, makes note that, under the conditions specified by him, raffinose is not only partly, but completely precipitated by ammoniacal lead acetate from its aqueous solutions. With sugar this is not the case.

Precipitation of Raffinose. Koydl.[3]

By the employment of a very great excess of reagents, or by several repetitions of the operation, raffinose is, in accordance with the statements of Weisberg, completely precipitated. With the directions given by the author for raffinose determinations (*i. e.* raffinose detection), the raffinose is not entirely thrown down in one precipitation, but will yield a sirup which allows the raffinose to crystallize direct and holds the sugar in solution; but for this weeks and months longer will be required.

Determination of Glucose by Means of Cupro-ammonium Acetate. Allessandri.[4]

The process is based upon the action of sulphuric acid upon cupro-ammonium compounds. Two solutions are employed in this method:

(1) Solution of normal copper acetate. Dissolve 19.45 grams of pure metallic copper in nitric acid and evaporate to dryness. The residue is dissolved in a little water and the copper is precipitated with caustic potash. The precipitate is brought upon the filter and is dissolved in warm dilute acetic acid. The solution and washings are evaporated to dryness on the water bath, and the residue, after dissolving in water, is made up to a bulk of two liters.

(2) The solution of sulphuric acid employed contains, per liter, 91.1 grams of monohydric sulphuric acid.

To test the relation of the two solutions to each other proceed as follows: Take 20 cc. of the copper solution, add 22 cc. water, and then run in, drop by drop, ammonia solution until the precipitate formed redissolves; then from a burette run in sulphuric acid until the precipitate formed disappears entirely, leaving a clear liquid. Ten cc. of the sulphuric-acid solution should correspond to 20 cc. of the copper-acetate solution.

To make a glucose determination 60 cc. of the copper solution are brought into a casserole, 10 to 20 cc. of the sugar solution to be examined (containing 5 to 10 grams of glucose per liter) are added, and the mixture is heated on a water bath about one-half hour. The contents of the dish are then filtered, washing well with boiling water. The filtrate is divided into three parts, each portion being titrated with ammonia and the standard sulphuric acid as before described.

The number of cubic centimeters of sulphuric acid required in the titration subtracted from the amount consumed in the titration of the copper acetate alone gives

---

[1] Oesterr. Zeit. Zucker-Ind., 1891, 20, 700; Jour. Soc. Chem. Ind., 1882, 463.

[2] Chem. Zeit. Rep., XVI, 1892, 14; Bull. Assoc. Chim., 1892, 7, 539.

[3] Oesterr. Zeit. Zucker-Ind., 1892, 21, 92; Chem. Zeit. Rep., XVI, 1892, 126.

[4] Le Stazione sperimentalie agr. Italiane; Bull. Assoc. Chim., IX, 556.

by the employment of the proper factor in the calculation the amount of glucose in the sugar solution under examination.

The Determination of Invert Sugar by Means of Soldaini's Reagent. Striegler.[1]

In the estimation of reducing sugars by this process, in products containing lime various reagents have been employed for the precipitation of the calcium. The carbonate, bicarbonate, phosphate, and oxalate of sodium have been separately tested, but without satisfactory results, the precipitant found most efficient being a solution of mixtures of sodium oxalate and bicarbonate. It is found that clarification of the liquid by means of blood carbon is quite advantageous, and it is claimed that reducing bodies other than invert sugars are absorbed thereby. The mode of procedure recommended in estimations with Soldaini's solution is as follows: Weigh 20 grams of substance, dissolve in water in a 200 cc. flask to a bulk of 140 to 150 cc.; add, according to nature of the product, 10 to 30 cc. of the oxalate-bicarbonate solution; heat to boiling, cool and make up to 200 cc.: shake with 1 to 4 grams of blood carbon and filter. Fifty cc. of the filtrate, together with 100 cc. of Soldaini's solution, are treated in the usual way. The following modified method of preparation of Soldaini's solution is recommended: Instead of 297 grams of $KHCO_3$, only 150 grams of this salt are employed, and in the place of the 147 grams there is used an equivalent weight of K_2CO_3 (101.4 grams). The two salts are dissolved in 600 cc. of warm (50°) water, and 100 cc. of an aqueous solution of 6.928 grams of copper sulphate are added thereto, the liquid being made up to 1 liter. In this way loss of carbon dioxid is avoided.

The Determination of Sugars with Ost's Cupro-potassic liquor. Schmoeger.[2]

The solution of cupro-potassium carbonate recommended by Ost[3] presents, as claimed by him, the following advantages over the solution of Fehling: Sucrose does not affect it; it can be preserved indefinitely; the duration of boiling has less effect upon the results; the extent of the reduction of copper is one and one-half to two times greater; the reduction factors of the solution are more distinctly marked for the various reducing sugars; and, in a general manner, the employment of the carbonate liquor is preferable to that of the tartrate solutions.

The author has attempted to verify the correctness of these claims and has found that, in general, they are well founded, save, perhaps, that the solution can be preserved for an absolutely unlimited period.

Ost's less concentrated solution is especially to be recommended for use in the determination of invert sugar contained in cane sugar in a state of impurity. There are some inconveniences, however, in the employment of the carbonate liquors when it is proposed to weigh the copper suboxid.

When raised to boiling in presence of a large excess of water carbon dioxid is expelled and cupric oxid is deposited, so that in the case of very dilute sugar solutions the cuprous oxid is brownish and contains cupric oxid.

Again, if the sugar solution contains a salt of calcium, a precipitate of calcium carbonate is formed.

This difficulty may be remedied by previously removing the lime by means of potassium oxalate.

Ost's solution,[4] above referred to, consists of 23.5 grams of $CuSO_4 + 5\ H_2O$, 250 grams K_2CO_3, and 100 grams $KHCO_3$ dissolved in water and made up to 1 liter. The solution, it is claimed, is especially adapted for gravimetric work, the copper being too slow of reduction for ordinary volumetric estimations. Fifty cc. of the solution are heated to boiling; 25 cc. of the sugar solution are then added, together with some water, and the liquid boiled for 10 minutes; then cooled, filtered through an asbestos filter, and the precipitate reduced in a current of hydrogen.

---

[1] Zeit. f. Rübenzucker-Ind., 1892, 459.

[2] Ber., 24, 3610; Bull. Assoc. Chim., VIII, 1892, 525.

[3] Ber., 23, 1035 and 3003.

[4] 1890, 3003; Chem. Zeit. Rep., 1890, 330.

Upon the Influence of the Presence of Lead Acetate upon the Determination of Invert Sugar According to the Fehling-Soxhlet Method. A. Bornträger.[1]

The author notes that in the determination of invert sugar by this method in solutions containing lead acetate the results obtained are invariably too low, and experiments were made by him to definitely determine the influence of the presence of both the neutral and basic salt.

The solution of the neutral acetate employed had a specific gravity of 1.223 and contained 36.5 grams of the crystallized salt in 100 cc., while the basic solution had a density of 1.25.

The invert-sugar solution used was prepared in the manner described by the author in a previous article and varying proportions of lead acetate were added thereto. The results of experiments with the solutions, both with and without lead acetate, are set forth in the following table:

Lead-acetate solution employed.....cc..	1	0.5	0.2	0.1	0
Invert-sugar solution required.....cc..	8.8	8	7.45	7.30	7.18
Invert sugar found.....per cent..	0.5852	0.6438	0.6913	0.7055	0.7173

It was noted that in the first experiment, instead of the beautiful red precipitate there was at first an orange yellow, becoming later a dirty red. In the second experiment the precipitate showed somewhat similar characteristics, while in the remaining tests the precipitates presented an almost normal appearance. In the first two experiments it was also observed that in testing for the end point of the reaction with potassium ferrocyanid and acetic acid perceptible quantities of ferrocyanid of lead were thrown down.

In the experiments with basic lead-acetate solution the invert-sugar solution used was prepared by the inversion of a sucrose solution in the cold.

The author notes that in a previous article he had shown that a normal sucrose solution to which one-tenth volume of 38 per cent hydrochloric acid had been added, after standing overnight, reaches the maximum of inversion.[2]

Nineteen grams of pure sucrose were dissolved in water, 10 cc. of HCl were added, and the solution was made up to 100 cc. and allowed to stand overnight. Twenty-five cc. of this solution were neutralized with caustic-soda solution, using litmus as an indicator, and the volume was then increased by dilution to 1,000 cc., yielding a 5 per cent invert-sugar solution.

Separate tests were made to determine what influence, if any, the presence of the litmus solution and sodium chlorid contained in the above solution had upon the precipitation of the copper in the Fehling-Soxhlet liquid employed, though with negative results. The author observed that in the presence of excessive quantities of litmus tincture the end reaction is not at all clearly defined, but with not more than 1 cc. of the litmus indicator per each 100 cc. of the invert-sugar solution no such trouble is experienced.

The results of the experiments with lead subacetate are given in the following table:

Lead-subacetate solution employed ...cc..	5	4	3	2	1	0.5	0.4
Invert-sugar solution used .....cc..	14.15	14.15	14.15	13.80	13.00	11.85	11.60
Invert sugar found.....per cent..	.364	.364	.3640	.3732	.3962	.4346	.4440
Lead-subacetate solution employed...cc..	0.3	0.2	0.15	0.1	0.075	0.05	0.0
Invert-sugar solution used .....cc..	11.20	10.80	10.70	10.60	10.48	10.42	10.30
Invert sugar found.....per cent..	.4598	.4769	.4813	.4858	.4914	.4942	.5

[1] Zeit. f. angew. Chem., 1892, 333; Neue Zeit. f. Rübenzucker Ind., XXVIII, 1892, 282.

[2] Zeit. Zuckers Ind. 1890, 876.

In the execution of these tests the dilution of the copper solution was so regulated that the total volume of the boiling liquid in every instance was about 60 cc.

As in the experiments with normal acetate, where large quantities of lead were employed (as in the first four tests), the copper precipitates presented quite an abnormal appearance, passing through various shades of color, from yellow orange to dirty red, while with the addition of only small proportions of lead solution bright-red precipitates were secured.

The results of these experiments indicate the necessity of complete removal of lead from solutions containing invert sugar previous to estimation of latter by means of reduction of copper.

For the precipitation of lead in sugar solutions the author recommends that sodium sulphate be employed in preference to the carbonate, as with the use of the latter the results are too high, while the sulphate is without influence upon the determinations.

With the addition of 5, 3, 2, 1, and .5 cc., respectively, of a saturated soda solution to 10 cc. of the copper liquid, instead of 10.3 cc. of the invert-sugar solution, there were required 10.1, 10.13, 10.15, 10.2, and 10.35 cc. to precipitate the copper.

Analysis of Sugars, Molasses, and Sirups. F. W. Babington.[1]

In the analysis of sugars clarification was effected by the use of 1 gram of bone-black and 20 to 30 cc. of alumina cream per each one-half normal weight of sugar.

In the execution of the optical inversion process the solution is heated in the water bath for 20 minutes and Wohl's figure of 142.7 is employed in calculating results. In the estimation of reducing sugars the amount of copper reduced was determined by weighing the precipitated suboxid, which had been previously brought upon an asbestos filter in a "Macfarlane milk tube," and thoroughly washed with hot water, alcohol, and ether. The tube and contents are then dried in a steam bath for about two hours, and the invert sugar is calculated from the weight of the Cu_2O. Water determinations in sirups were performed by saturating asbestos fibre, contained in a milk tube, with about 20 cc. of 20 per cent solution of the sample and drying the tube and contents for about forty-eight hours at a temperature of from 90 to 100°.

Determination of Sugar in Cocoa Preparations. Schroeder.[2]

Determinations of starch sugar in chocolates, etc., can not with certainty be made by means of simple reduction of copper solution, as it has been found in certain experiments that the results of reduction tests were increased (0.24 to 0.27 per cent) by the reducing sugars contained in the commercial cane sugar.

The inversion method admits of the surer tests for greater quantities of commercial glucose. The lowering of the rotation of the solution consequent upon the admixture of glucose is a good indication of the presence of that substance. The estimation of the dextrin in the solution after fermentation with yeast is also another means of determining the existence of commercial glucose in the preparation.

Boyer's Method of Ash Determination in Sugars with Benzoic Acid. Paczosski.[3]

The author shows that by means of this method there can be secured results which are useful and are comparable with each other, but the process is not generally employed nor do the results obtained agree with those of Scheibler's method.

As Weisberg rightly remarks, as against Alberti and Hempel's new ash method with quartz sand, the remaining processes are not to be taken into consideration.

Determination of Ash in Sugar Products. Donath and Hattensaur.[4]

The authors have attempted to determine the ash in raw sugar, molasses, etc.,

---

[1] Analyst, October, 1891, 181.

[2] Zeit. f. angew. Chem., 1892, 173; Chem. Zeit. Deutsche, 1892, 134.

[3] Bull. Assoc. Chim., 1892, 9, 612; Chem. Zeit. Rep., 1892, XVI, 48.

[4] Chem. Zeit., 1891, xv, 520; Jour. Soc. Chem. Ind., 1891, x, 781.

directly, by mixing the sample with a weighed quantity of powdered pumice, ignited alumina, magnesia, or copper oxid previous to combustion, but without satisfactory results.

Better results are obtained by first carbonizing the sample, then heating at the lowest possible temperature in a mixture of air and oxygen, and finally in pure oxygen. The ignition is best conducted in a platinum crucible, with a small, shallow platinum dish with perforated bottom fitting inside and covered by a perforated lid.

The perforated dish serves to distribute the entering gas, which would otherwise act more energetically upon the substance in the center of the dish.

Estimation of Ash in Raw Sugar. A. Stift.[1]

Having used Grobert's method of burning to ash raw sugars and sugar liquors with the aid of oxalic acid, the author finds that the results obtained coincide with those obtained by burning the ash alone. The proportion of oxalic required is, however, in most cases very considerable, varying from twice the weight of the sugar in the case of raw sugars to five times the weight of substance in the case of molasses. To use less oxalic acid by sprinkling more upon parts of the ash which have not burned perfectly has not proved, in the author's hands, an altogether successful modification.

Determination of Ash in Massecuites.[2]

Take a volume of solution corresponding to 5 grams of the product, evaporate in a weighed platinum capsule, and then incinerate with H_2SO_4. For second jet products, a little refined sugar should be added to facilitate incineration.

New Method of Determination of Ash of Raw Sugars and Molasses. By Alberti and Hempel.[3]

The disadvantages, attendant upon the employment of the usual method of incineration with sulphuric acid are referred to, while the substitutes hitherto proposed are not considered at all satisfactory.

The method proposed by the authors depends upon the incineration of sugar or molasses, previously mixed with pulverized quartz, 6 to 7 grams of the latter being employed with 5 grams of sugar.

The platinum capsule containing the mixture is placed in a muffle and immediately heated with a full flame. The sugar burns without frothing, and at the end of from one-half to one and a quarter hours, according to the nature of the prduct, the incineration is completed, unless it becomes necessary to stir the mass.

The principle of the method is this: In the presence of silicic acid the incineration is complete, without charred residue; it forms silicates instead of carbonates, the silicic acid being substituted for the organic acids already present. The sulphates and alkali chlorids already contained in the sugar are not decomposed, at the temperature of the muffle, by the silicic acid, and rapid and reliable determinations of the inorganic constituents of the sugar can thus be made.

The authors have made a large number of comparative tests with the new quartz method and the ordinarily employed sulphuric-acid process.

The Determination of Ash According to the Method of Alberti and Hempel. Sidersky.[4]

The author reviews the various methods heretofore proposed or employed in ash determinations, and notes the desirability of obtaining in the ash the inorganic compounds in their original forms of occurrence. Alberti and Hempel claim that their quartz-sand method yields an ash in which the mineral salts have undergone no change.

---

[1] Oester Zeit. Zück. Ind., 19, 484; Jour. Chem. Soc. (Abstr.), 1891, 1297.

[2] Report of Commission of "Assoc. des Chimistes de Sucrerie;" Jour. des Fabr. de Sucre, XXXII, 1891, 34.

[3] Jour. des Fabr. de Sucre, XXXII, 1891, No. 34.

[4] Journal des Fabr. de Sucre, XXXII, 1892, No. 37.

This conclusion is drawn from a large number of experiments, in which pure sugar, mixed with varying proportions of the chlorids and sulphates of potassium and sodium, was incinerated, the salts added being found to suffer no loss in weight.

It is remarked, however, that the question of nitrates has been overlooked, this class of salts being completely decomposed in the presence of quartz.

Numerous dark sugars which have been examined contain quite appreciable amounts of nitric acid (from 0.04 per cent to 0.32 per cent), and this acid is completely driven off by the silica, the weight of the ash obtained being diminished to that extent.

The method with quartz sand, however, is extremely easy of execution, and the burning to ash takes place very rapidly.

A low-grade raw sugar was tested as to ash content according to the new method and sulphuric acid, chlorin and nitric acid being determined both before and after incineration. The quantity of sulphuric acid and chlorin present originally showed only a slight decrease on completion of the incineration, while the nitric acid (18 per cent) had been completely expelled.

If it is shown by a large number of determinations that the sulphates and chlorids undergo no change in the burning to ash of the sugars in presence of quartz, then a separate determination of the nitric acid, followed by the estimation of the ash, will give the real amount of mineral substance contained.

An objection to the general employment of the Alberti and Hempel method is on account of the change in the formula for calculation of the "rendement" which will be made necessary by its introduction.

The Determination of Ash in Sugars. Alberti and Hempel.[1]

In replying to the communication of Sidersky upon this subject (Jour. Fabr. Sucr., XXXII, 37), it is stated that the question of the influence of the quartz sand upon the nitrates had not been overlooked by the authors in the proposal of their method of ash determination. The fact that nitric acid is expelled by heating in contact with the silica is considered to be rather of advantage in ash determinations, as, in the strictest sense, it is not believed that nitric acid should be classed with the inorganic matters of the sugar.

It is admitted that the new method, from a theoretical standpoint, is not in all cases absolutely free from slight error, but, considered practically, it possesses many advantages over the older methods.

Sidersky's observation as to the change in the "rendement" formula which would be necessitated by the use of this method is not thought worthy of serious consideration, in view of the plan already under discussion with regard to a change in the manner of calculation of the "rendement."

It is remarked that the fears first entertained by the authors with regard to the deleterious effect of the quartz sand ignitions upon the platinum vessels were without foundation.

After forty determinations it is found that by treating the dishes with fused soda, followed by thorough scouring, only a slight loss in weight is observed.

Determination of Ash in Sugars.[2]

Sidersky takes issue with the statement of Alberti and Hempel (Jour. Fabr. Sucr., No. 39), with regard to the exclusion of nitrates from among the inorganic constituents of sugar products and states that he fails to understand why any similarity in mode of formation of nitric acid in nature to that of organic substances should be taken as a ground for a failure to class nitrates among the mineral matters.

Determination of Ash in Sugars.[3]

The Chemiker Zeitung, in referring to the proposition of Sidersky with regard

[1] Jour. des Fabr. de Sucr., XXXII, 1891, No. 39.
[2] Jour. des Fabr. de Sucre, XXXII, 1891, 40.
[3] Jour. des Fabr. de Sucre, XXXII, 1891, 43.

to making separate determinations of nitric acid in connection with the Alberti and Hempel method, observes that the French and possibly the Belgian sugars contain larger quantities of nitrates than do the German and Austrian products, so that, on this ground, in the latter countries there is not the same objection to the employment of this method in ash determinations.

As the new method gives accurately the proportion of inorganic constituents, and, by difference, that of the organic matters, it affords an excellent basis for calculating the commercial value of sugars.

Alberti and Hempel's Method of Ash Determination. Donath and Eichleiter.[1]

The authors have determined that in the reduction of sugars or sugar products to ash with quartz, the chlorids and alkali sulphates were not decomposed, though as soon as these salts are heated alone with quartz not inconsiderable losses occur. The method is thus, for practical purposes, entirely useful and commendable and gives quite good concordant results.

With regard to the nitrates (as the Chemiker Zeitung had previously observed) for Austrian and German products this point need not be considered.

It is certain that the new method of ash determination enables the inorganic constituents of the sugars to be determined more accurately than by any process previously known, and it is possible, which appears still more important, to obtain the quantity of organic nonsugars with very approximate correctness. It is observed that the wearing of the platinum vessels is always very noticeable, on which account it is preferred to perform the ash determinations in porcelain dishes of a corresponding form.

Determination of Ash by Means of Ferric Oxid.

Courtonne[2] reports the results of some practical tests in ash determinations by the substitution of oxid of iron for silica. The results are practically the same, but the oxid of iron presents many advantages over the silica, the principal one of which is that the incineration proceeds with very great rapidity, 20 minutes, at the most, being sufficient to complete the process either with sugars or molasses. Another advantage is that the platinum vessels employed are not attacked, as is the case with the use of the Alberti and Hempel method.

Quick Method for the Determination of Water in Massecuites, Molasses, etc. H. Pellet.[3]

It is noted that in solutions of pure sugar the proportion of dry substance present can be readily determined by merely taking the density of the liquid, and on referring to tables the desired figures can be obtained. Massecuites, or sirups of pure sugar, when dissolved in definite quantities in a known volume of water thus give data from which the proportion of water originally present can be easily obtained. The author notes, however, that solutions containing equal weights of sugar and of the dry matter of massecuites of first jet possess quite different densities. For instance, 50 grams of pure sugar dissolved in water and made up to a bulk of 250 cc. give a density at 15° of 1.08354, while 50 grams of a dried massecuite (of first jet) dissolved in a like manner give a solution with a density of not more than 1.0788.

Thus it is that by taking the density too much dry matter is indicated, and the coefficient of purity varies according as the water content is obtained from the density or is actually determined. The author cites an example where the apparent purity (by density) is from 88.5 to 90, while the real purity is 90.1. He gives also an experiment of Sidersky where a massecuite of second jet gave an apparent purity of 81.1 and a real purity of 84.2, the water content calculated from density being 9.75 per cent, while the actual determinations showed 13.7 per cent.

---

[1] Oester, Zeit. Zucker-Ind. 1892, 21, 281; Chem. Zeit. Rep., 1892, XVI. 198.

[2] Bull. Assoc. Chim., 1892, 806.

[3] Jour. des Fabr. de Sucre. XXXII, 1891, No. 38.

Since for rapid work the method of actual determination, either in the air bath or in vacuo, requires too much time, a mode of procedure involving little time and labor was desired.

The difficulties referred to were avoided by the construction of tables derived from actual experiments with different grades of factory products, a separate table being prepared for each class of products. For example, the water content of a massecuite is determined by the usual drying process; then there are next weighed up such quantities of the massecuite as will give weights of solid matter corresponding to massecuites having water contents varying from 0 to 15 per cent, 50 grams being in each case used as a basis from which to calculate the weights required. The quantities of massecuite thus weighed up are next dissolved in water, the solutions made up to 250 cc., and the densities carefully taken at 15°. There is in this manner obtained a series of densities corresponding to certain definite proportions of the solid matter contained in the massecuite, and the data being plotted on paper interpolations can be made graphically, giving the water content in half per cents.

Experiments can, in addition, be made with other representative samples, thus rendering the tables more trustworthy, and still other tables can be constructed for molasses and other liquid products.

It is claimed that, when practically applied, this method gives a water content closely approximating that obtained by actual determination, the differences in many cases being less than from one-fourth to one-half per cent.

The Determination of Water and the Organic Constituents of Raw Sugar. Alberti and Hempel.[1]

As Soxhlet has already shown, the common air bath is, in many respects, imperfect and faulty. For raw sugar it is to be noted especially that with temperatures of more than 100° to 110° slight decompositions may occur, and these temperatures in the ordinary drying chambers are often considerably exceeded, even when the thermometer in the center indicates only 105.° With Soxhlet's and Schwackhöfer's apparatus, however, the authors have always obtained good and reliable results. Nevertheless, they desired to employ an apparatus of newer and better construction, combining the advantages of both of those mentioned with the employment of a saturated salt solution (boiling point 107°) as a heating material, and with a capacity for drying many samples (up to 25) at once at a constant heat of 100 to 100.5° within two or three hours with reliable and concordant results.

Special parallel experiments show that with the employment of 10 grams the determinations are exact to 0.1 per cent, so that the method satisfies the requirements of commerce.

The determination of the inorganic constituents according to the quartz-sand method is performed by the authors with the employment of 10 grams and the results are equally exact. Gas which contains much sulphuretted hydrogen or carbon disulphid, and produces, on combustion, oxidation products of sulphur, may be the cause of considerable error, since the quartz-ash absorbs sulphuric acid. Care should be taken, then, to exclude such products as completely as possible from contact with ash experiments.

The authors do not anticipate the immediate general employment of the new method for commercial purposes (chiefly on account of the present method of calculating the "rendement"), but hope that the grounds of opposition to its use will soon disappear.

Determination of Water in Raw Sugars.

Stift, in his report to the Austro-Hungarian Association of Sugar Chemists[2], notes that even at 100° he has observed that quite perceptible decomposition takes

[1] Zucker—Ind., 1892, 17, 385; Chem. Zeit. Rep., 1892, XIV, 106.
[2] Chem. Zeit., 1892, XVI, 923.

place, and found that, in one instance, after six hours' drying there was a decrease of 1 per cent in polarization.

In alkaline products it is found that ammonia is given off, while acid products on long heating evolve volatile fatty acids. The results obtained with Soxhlet's drying chamber were about 1 to 2 per cent higher than with Schwackhöfer's vacuum apparatus. The position of the samples in the drying chamber is said to affect the results to no inconsiderable extent, five control determinations upon same substance showing extreme differences of .3 per cent.

The Determination of the Water Content of Raw Sugars and Sirups. By A. Herzfeld.[1]

In order to execute as accurately as possible the determinations of water in raw sugars and sirups, and with the exclusion of oxidation products, the vacuum drying apparatus was tested. It was impracticable to employ a larger size vacuum drying chamber on account of the difficulty of constantly maintaining a vacuum by means of a water aspirator, and a small Scheibler vacuum drying apparatus was so remodeled as to be adapted to the requirements of the work.

The apparatus was provided with drying cylinder, condenser, manometer, etc., and was so arranged that the drying chamber was heated by direct contact with boiling water. By a comparison of the results of the determination of the water content of a sirup in this apparatus and in a good air bath provided with double walls there were always, with the vacuum apparatus, several tenths per cent less water found than with the air bath at 105°.

The results also were not perceptibly changed when a layer of lime, for the absorption of water, was introduced in the vacuum drying chamber. With a sirup prepared from pure sugar, pure salts, and a known quantity of water the same behavior was shown, the air bath giving, in this case, the correct per cent of water. In order to raise the temperature of the vacuum drying bath above 100° there was employed instead of water, as a heating liquid, toluene, having a boiling point of 109° to 110°, and by means of which the interior of the drying chamber attained a temperature of 107° to 108°. Water determinations were then made as follows: First, in vacuum at 90° to 95°; second, in vacuum at 107° to 108°; third, in air bath at 105° to 108°. Experiments were made upon sound, invert-sugar-free factory sirups, with slight alkaline reaction, and also upon weakly acid impure refinery sirups containing invert sugar.

1. Water determinations in sound sirup.

Sample.	Vacuum 90° to 95°.	Vacuum 108°.	Air bath 105° to 108°.
No. 1	19.95	20.10	19.96
No. 2	20.17	20.38	20.25
No. 3	.....	15.15	15.10
No. 4	.....	15.21	15.02
No. 5	.....	15.64	15.56

2. Water determinations in sirups containing invert sugar.

Sample.	Vacuum 90° to 95°.	Vacuum 108°.	Air bath 105° to 108°.
No. 1	25.00	25.28	25.59
No. 2	22.90	23.21	23.55

The results show that although at 95° in vacuo a constant weight is secured, still on raising the temperature to 108° a marked decrease takes place, and with the sound

[1] Zeit. des Ver. f. Rübenzucker-Ind., 1892, 257.

sirups the water content is, throughout, slightly higher than that obtained by the use of the air bath. The author notes that in the vacuum the drying takes place much more readily and more perfectly where the sirup is mixed with a considerable quantity of sand, complete drying being practically unattainable in the absence of sand.

The unsound refinery sirups show a loss of weight in the air bath several tenths of a per cent in excess of that obtained in the vacuum at 108°, indicating that oxidation of such products takes place very readily in contact with air at that temperature.

For securing correct results in water determinations the author considers it essential to perform the drying in vacuo at a temperature slightly above 100°.

For the attainment of a satisfactorily high constant temperature toluene is highly recommended, chiefly on account of the fact that it does not attack the metal of the apparatus as does a salt solution, though on account of its easy inflammability it should be used with great caution.

Where steam is constantly to be disposed of, it can be advantageously used for heating the drying chamber.

In the determination of water in raw sugars the important differences noted above were not exhibited. With drying in vacuo at 95° the same results were obtained as in the air bath or vacuum at 108°. The author observes that with the purer products the quantity of hygroscopic substances is so small that scarcely appreciable differences are noted in drying by the various methods alluded to.

Determination of Water in Massecuites.[1]

The water is determined with a volume of solution corresponding to a definite weight of massecuite (1 gram for example).

The drying is performed in a drying chamber at 110° until a constant weight is secured.

It will be better, however, to employ in the drying operation the apparatus of Courtonne (with vacuum), a drying chamber supplied with a current of hot air, or the vacuum-drying apparatus of Vivien.

Pellet's Continuous Polarization Tube.

Lohr[2] has found this tube, which he has improved in quite an advantageous manner, quite worthy of commendation.

When water is contained in the tube, 20 to 30 cc. of a low —40 cc. of a high polarizing sugar solution are required for total displacement. The tubes thus improved should be quickly introduced into general use, as they are quite accurate and simple in their operation.

Pellet's Continuous Polarization Tube. Carlier.[3]

This tube has been thoroughly tested, being used largely in the testing of beets for selection of seed, and has been found to give exact and trustworthy results.

It is stated that with a single apparatus one can make six to seven observations per minute; 3,600 to 4,200 in ten hours.

A Modification of Pellet's Continuous Polarization Tube. Stift.[4]

The author describes a slight modification proposed by Strohmer, which allows the tubes to be filled, under all circumstances, free from air bubbles, and polarization can be made immediately after the displacement of each solution by the following:

The saving of time, although amounting to only ten seconds for each operation, is, on the whole, very considerable.

For example, Brien, at the Leopoldsdorf beet-culture station, was able to make and note, on an average, 110 polarizations per hour, filling the tubes himself.

[1] Report of Commission of "Assoc. des Chimistes de Sucrerie," Jour. Fabr. de Sucre, XXXII, 1891, 34.
[2] D. Zuck. Ind. 1892, 17, 230; Chem. Zeit. Rep., 1892. XVI, 90.
[3] Sucr. Belge, 1892, 20, 238; Chem. Zeit. Rep., 1892, XVI. 106.
[4] Oester. Zeit. Zucker-Ind., 1892, 21, 265; Chem. Zeit. Rep., XVI, 1892, 198.

New Areo-Picnometer for Determining Specific Gravities of Juices, Sirups, etc. Fristch.[1]

The apparatus consists of a flask of 50 cc. capacity, attached to the lower part of a spindle, whose construction is somewhat similar to that of those ordinarily employed in sugar work. The flask, after being detached, is filled with the liquid under examination and is again connected with the spindle, the apparatus then being placed in a vessel of distilled water.

Both the degrees Baumé and the absolute weight of the liquid in the flask can be read from the graduations on the stem of the spindle. As each degree Baumé on the scale extends over about 28 millimeters, great accuracy is claimed for the apparatus.

A New Picnometer for Sirups.[2]

Neumann proposes a new picnometer, resembling somewhat in form an Erlenmeyer flask, the bottom of which (viewed exteriorly) possesses a considerable degree of concavity. By means of this a quick and uniform cooling, it is claimed, can be secured.

The upper rim of the picnometer is ground smooth, and after the sirup has cooled any excess of it remaining above the rim of the flask can be scraped off with a spatula.

Tests for Certain of the Carbohydrates. B. Tollens[3] and others.

The formation of levulinic acid on boiling a substance with hydrochloric acid, sp. gr. 1.09 to 1.10, for twenty hours is considered as strong evidence of the presence of a true carbohydrate.

In the case of dextrose, or of such substances as lactose, raffinose, etc., which yield dextrose, the presence of this sugar can be easily proved by oxidizing the substance with nitric acid, the saccharic acid formed under these conditions being a proof of the presence of dextrose or of dextrose groups.

The detection of levulose or of levulose groups in vegetable products is extremely difficult. The ready decomposition of this sugar by heating with sulphuric or dilute hydrochloric acid yielding brown solutions may serve, however, to distinguish it from dextrose.

Alkali Determinations in Dark-Colored Sugar Products.[4] Buisson.

Twenty-five cc. of the liquid to be examined are brought into a flask, together with a drop of an exactly neutralized rosolic-acid solution and 10 cc. of pure neutral ether. The titration is then performed with sulphuric acid, the liquid being well shaken after each addition of acid and the ether being allowed to separate. As soon as the reaction is completed, that is, when the alkali becomes saturated, the rosolic acid is liberated and dissolves in the ether, coloring it strongly, so that a distinct and instantaneous change results.

Sampling and Preserving Juices.[5]

In order to preserve samples of diffusion juices Sidersky recommends that the juices be at once poured into vessels containing solid subacetate of lead, the mixture being thoroughly shaken after each addition of juice.

Mr. Ross. I suppose it will save time if the recommendations are acted upon just as read instead of going over them and reporting them.

Mr. Ross read the first recommendation.

Mr. Wheeler. I think the method recommended here is extremely

[1] Oester. Zeit., Zucher-Ind., 1891, 268; Bull. Assoc. Chim., 1892, 551.
[2] Zeit. f. anal. Chem., 1892, 63.
[3] Landw. Versuchs-Stat., 39, 401; Jour. Chem. Soc. Abstr., 1892, 249.
[4] Chem. Zeit. Rep., xvi, 1892, 76.
[5] Jour. des Fabr. de Sucre, xxxii, 40.

unreliable, viz, the drying of material in an open dish. The method that we regard as the most reliable is the drying in vacuum. Now, I recommend that the reporter next year determine the relative value of drying in vacuum as compared to drying in an open dish, and of determination of moisture. I make that motion.

Seconded.

Mr. WILEY. I would like to have Mr. Spencer state briefly the method and apparatus used by him.

Mr. SPENCER. In the experiments we made we had considerable difficulty in water accumulating in the drying tube. At first we used the method described in the book. Afterward we modified that, using a tube with a small bulb, and in connection with that we used an ordinary water pump, and had no difficulty in producing a vacuum of 27½ or 28 inches. The drying is complete in about ten hours. The principal difficulty we found was in preventing the moisture from collecting in the upper part of the drying vessel, but this is avoided by inserting a catch bulb, which prevents any of the water from dropping back into the tube.

The PRESIDENT. The motion is that the reporter be requested to investigate the merits of drying in vacuum and in air.

Adopted.

The PRESIDENT. Now we come to the next recommendation.

Mr. ROSS. I do not make any recommendation—any changes—under that head on that page. While under that head I will make a recommendation that the reporter for the ensuing year report upon the determination of water from density by the table by Pellet, which method is described in one of the abstracts which I have made.

Adopted.

Mr. ROSS. On page 226, before we leave the subject of ash, I recommend that the reporter for the ensuing year be instructed to test the method proposed by Alberti and Hempel.

Adopted.

Mr. ROSS. On page 226, under the head of "Reagent, alkaline copper solution," I recommend that the figures 500 be employed instead of 1,000, as those are the figures almost universally employed. It is unnecessary to read this, as it is in the report, and there it is much more accurate in statement.

Seconded.

Mr. WILEY. There are one or two typographical errors in the methods which are given on the official report here. Anyone can see they are errors, but I have deemed this matter of sufficient importance to look into it. Mr. Ewell has been working a long time on the subject and very carefully. Now, I wish to offer an amendment—a substitute—which is too long to read, but it embodies essentially the methods here, but more compact and in a more reliable form. I do not wish these to be adopted as official methods, only as provisional methods, for I do not

believe any methods ought to be adopted as official until they are thoroughly tried. I beg to submit to the reporter this substitute for the methods for determining reducing sugars with the recommendation that if it be accepted by him it be adopted provisionally for use by the members of the association.

Mr. EWELL. According to the method originally used in the laboratory of the U. S. Department of Agriculture for the electrolytic determination of the copper obtained in reducing-sugar estimations, the precipitate of cuprous oxid was dissolved in nitric acid and heated with sulphuric acid until all the nitric acid was removed. Eight or ten drops of concentrated nitric acid were added and the liquid transferred to a platinum dish for electrolysis. This process insures the presence of enough nitric acid to give a bright deposit of metallic copper, and avoids so great an excess as would require special precaution in washing. When it is desirable to avoid the long evaporation the precipitate is dissolved with 30 cc. of a dilute mixture of nitric acid and sulphuric acid (65 cc. of sulphuric acid, sp. gr. 1.84, and 50 cc. of nitric acid, sp. gr. 1.42, per liter), and the solution filtered, and washed into a platinum dish. This saves the long time required for the evaporation, but of course the use of so much nitric acid necessitates great care in washing the deposit of copper. It has been found in our work in the laboratory of the Department of Agriculture that the use of so much nitric acid is rendered safer if the first washing of the deposit be made with water slightly acidulated with sulphuric acid. This insures a good current until the greater part of the nitric acid has been removed.

The PRESIDENT. Does the reporter accept it?

Mr. WILEY. It was prepared by Mr. Ewell, and I think we can rely upon it as the best way possible at the present time. It is so like the one in the book that it is hardly worth while to have it read.

Mr. ROSS. I accept it in lieu of the methods as printed in last year's bulletin.

Adopted.

Mr. ROSS. I was going to suggest a modification in regard to the electrolytic determination. Under the head of "Gravimetric method" I would like to have it read, "dilute until the quantity of nitric acid is not in excess of 5 per cent and reduce without using sulphuric acid." It will avoid a considerable time in the evaporation to expel the nitric acid.

Mr. WILEY. This is such a novel innovation that before we adopt it I think we had better have it studied for another year. I therefore move that this recommendation be referred to the reporter for next year, and that he be instructed to investigate this method.

Mr. ROSS. I am perfectly willing to accept it that way. But I think it is not entirely novel. Two years ago I tried a process essentially the same.

Mr. WILEY. It is certainly novel in sugar analysis so far as the proc-

esses in this country are concerned, and, for that reason I would like to see it further investigated without adopting it as a permanent method, but as a provisional one.

Mr. Ross. I am willing to let it go at that.

Mr. Wiley. The method will certainly save time. I am very much impressed with the idea that this will be a very good thing.

The President. The motion is to adopt it as a provisional method.

Adopted.

Mr. Ross. Under the head of "Sucrose," I have prepared something which is hardly worth while to read. It is the same as published in the Eighth Annual Proceedings.

Mr. Wiley. I move that the recommendation be accepted and inserted in the method.

Carried.

Mr. Ross. On pages 232 and 233, under the head of "Method of Clerget," I recommend, in addition to that, the method which is placed under the head of "Elective," which was adopted as the method of the association two years ago, be continued. It has been tried in a laboratory where the heating was continued to fifteen minutes at a temperature of 67° to 70° and when the factor 142.45 was employed. The temperature makes quite a considerable difference in the rotation, but after fifteen minutes it is very much less than at ten minutes.

Mr. Scovell. I understand that on page 233 the reporter substitutes the factor 144 instead of 142.4.

Mr. Ross. That we have already adopted.

Mr. Wiley. Possibly the time given here secures a longer heating than is necessary.

Mr. Ross. If this is a provisional method I do not think any harm can come from it.

Adopted.

The President. Shall we take up the next report or take a recess?

Mr. Scovell. I move that the rules be suspended and that we proceed to the report on fermented liquors.

Adopted.

The President. I would like to state that it is absolutely necessary that every one of the reporters have his report in shape and presented to the secretary before he leaves the city.

I call on Mr. Crampton to read his report on fermented liquors.

Mr. Crampton. My report is as follows:

REPORT ON FERMENTED LIQUORS.

By C. A. Crampton.

The work of the association upon this subject has been confined to printing a translation of methods adopted by European organizations, with some slight changes each year based upon personal interviews of the reporter with prominent chemists of Germany.

It appeared to be time that the custom of actual comparison of results, which has been so productive of good in the other classes of substances covered in the work of the association, should be applied to this as well.

The "methods of analysis of fermented liquors" seem to have resolved themselves into methods for wine analysis, beer analysis having been eliminated. Wines are of the greatest agricultural interest and doubtless deserve the largest share of attention at the hands of this association.

Accordingly I decided that the first analytical work should be done on wines, and sent out two samples, one dry and one sweet wine, to analysts, asking for determinations of alcohol and total solids only. The samples were drawn from the same receptacle, so that the trouble experienced in other branches of the association work resulting from incomplete mixing of samples could not obtain here. The samples may have undergone some change, of course, before being examined, but this I think hardly likely; certainly not in the case of the angelica.

The samples were accompanied by the following letter of instructions:

TREASURY DEPARTMENT,
OFFICE OF THE COMMISSIONER OF INTERAL REVENUE,
Washington, D. C., June 2, 1892.

DEAR SIR: In accordance with your letter of acceptance in reply to my circular letter of April 25, I sent you yesterday, by express, a box containing two samples of wine.

The "claret" is a dry wine, the "angelica" a fortified sweet wine.

You can use the methods prescribed on page 204, Bulletin 31, for the determination of alcohol and of "extract" (total solid), or such modifications as you think best.

It is more accurate to weigh out the amount taken for distillation and the distillate than to measure each at the same temperature, and I would advise this modification. If it is measured out the specific gravity of the wine must be taken, especially of the sweet wine, in order to calculate the percentage by weight. Return the percentage, both by weight and by volume, using the alcohol tables given in Bulletin 31.

For the extract not over 10 grams of the sweet wine should be weighed out, best from a flask or into a dish. A determination of the solid matter by evaporation, making up and taking the specific gravity by means of a Balling or Brix spindle, would be very desirable.

In order to best preserve the samples before and during analysis, they should be laid on their sides in a cool place. As this is the first time such samples have been sent out to association members it is very important that whatever methods be used they should be fully described, and I urge you to give a complete and detailed description of the method you use and the complete calculation of your results. Results should be sent in not later than July 1.

Should your samples be lost or broken in transit write immediately for another set.

Very respectfully,

C. A. CRAMPTON,
Reporter on fermented liquors.

Reports were received from fourteen analysts, whose results on alcohol and total solids are given in the following table:

Analyst.	Claret.		Angelica.	
	Alcohol, by weight.	Total solids.	Alcohol, by weight.	Total solids.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
C. L. Penny (Delaware)	9.92	3.36	15.30	17.44
G. H. Failyer (Kansas)	13.50	3.13	16.10	16.34
C. B. Collingwood (Arizona)	8.47	3.03	14.77	16.50
T. S. Shiver (South Carolina)	10.63	2.99	15.99	15.66
R. C. Kedzie (Michigan)	10.67	3.10	*16.87	16.76
George E. Colby (California)	10.61	3.08	15.98	15.65
E. N. Eaton (Iowa)	10.30	2.93	15.30	15.45
Duncan Adriance (Texas)	13.80	3.25	†21.44	17.87
H. A. Huston (Indiana)	10.14	3.22	15.19	16.52
B. W. Kilgore (North Carolina)	10.35	2.84	15.63	15.79
George L. Teller (Arkansas)	10.17	2.90	15.53	15.90
Oma Carr (United States Department of Agriculture)	11.17	2.69	18.59	17.36
A. E. Knorr (United States Internal Revenue)	10.43	3.28	15.66	17.38
C. A. Crampton (United States Internal Revenue)	10.52	3.10	15.75	16.81
General average	10.44	3.06	15.89	16.53
Difference between highest and lowest	5.03	.52	3.82	2.42

*Results calculated by reporter from a temperature of 23° as made to 15.5°.

†These results are omitted from the averages; they appear to have been calculated at a higher temperature, and the data accompanying them are not sufficient to enable the reporter to make a recalculation of them.

In the averages one set of results was omitted entirely because they seemed to have been made at a high temperature and calculated out without correction, although no distinct statement was made to that effect. Another set was recalculated and the results included in, as the analysts' accompanying report stated that the estimates were all made at a temperature of 23° with no corrections for temperature.

The quantities taken for the alcohol determination were weighed instead of measured by all the analysts except in Michigan, Iowa, and the United States Department of Agriculture. The Texas report does not state whether actual weight or volume corrected by specific gravity was used.

Taking into consideration the fact that these determinations are out of the line of the work of most of the analysts, together with the inherent difficulties of the estimations themselves, particularly those of the alcohol, it seems to me that these results are remarkably good. There is little doubt but that the aberrant results on alcohol in some cases are due to errors in the calculation of results, especially with reference to specific-gravity figures. I have often had occasion to observe the looseness of chemists generally, and of chemical literature as well, in the calculation and expression of specific gravities. In two cases above it was evident that such error had been made, and there is every reason to believe that some of the other wide variations were due to the same cause, though not evident on the face of the reports. If two or three of these extreme cases were excluded the rest would be brought, in the alcohol determination, to a range of 1½ per cent, which is fairly comparable with the work of the association in other lines; and, without excluding any of the results, the extract determination is fully equal to potash and phosphoric-acid work, which is quite remarkable in a new subject. It is scarcely necessary to point out, however, that there is a wide margin for improvement with a range of over 5 per cent in the most important determination to be made in wines.

Most of the analysts sent detailed descriptions of their manipulations and complete data for the calculations of results.

These descriptions follow in a more or less condensed form:

KANSAS.

In the case of the total solids longer heating caused greater loss, and these weights represent between three and four hours' heating instead of the two and one-half suggested. You say to fully describe the method and "the complete calculation." In the latter case I suppose the results of the completed calculation will be sufficient if the complete method is given, as clerical work only would be involved.

I have been accustomed to weigh samples of liquors for analysis, hence did not measure nor take the gravity of the wine.

Briefly, I weighed from a stoppered flask about 100 grams of the wine, weighing correct to milligrams. It was diluted in the flask from which it was to be distilled with 50 cc. distilled water. The distillation was from a sand bath, the condensing water escaping at a temperature between 17° and 18°, never beyond these limits, and for the most part about midway between. The distillate was collected in a stoppered flask, and the operation stopped when a little over 100 cc. had come over. The gravity was taken at 15.5° in a 100-gram flask with thermometer and overflow space. From Table III, Bulletin 31, the per cent by weight was found. This multiplied by the weight of distillate and the product divided by the weight of wine taken gives the per cent on the original liquid.

I have not given the per cent by volume, since the wine was not measured and the table can not be used for the purpose directly.

I think it a mistake to use 15.5°. It is said to be the "world" method. Unless it has intrinsic merits it should not be retained. There may be other temperatures better than 25 degrees, but I object to one so low as 15.5°. You must have experienced the difficulties and I will only hint at them. It is more difficult to maintain a uniform temperature throughout, the greater its difference from that of the work room. In our summer weather weights can not be taken at 15.5° owing to the condensation of moisture. If the flask be allowed to attain the temperature of the room time is lost, and what is of much greater importance where there is so great a change the expansion is great and the perfection of the flasks with which I have had to deal has not been such as to provide entirely against loss when the liquid expands.

ARIZONA.

Claret.—The specific gravity of wine taken by picnometer at temperature of laboratory, sp. gr. of distillate, picnometer at 15.50, wine, 0.9936; distillate, 0.9858.

Total residue: Evaporated a weighed quantity (9.6000 grams) in platinum dish over water bath; dried in oven at 100° for two and one half hours. Weight was not reduced by heating in oven.

Alcohol: Distilled 98.8820 grams with 50 cc. water; distillate weighed 98.21 grams. Cooled to 15.5 Sp. gr.=.9858; found alcohol equivalent in table to be by weight 8.61; by volume 10.70. As the distillate was 0.9928 of the amount taken, then alcohol by volume=10.70 × 0.9928 = 10.52 per cent; alcohol by weight = 8.61 × .9928 = 8.47 per cent.

Total acid: Titrated 10cc. wine against standard NH_4OH; determined neutral point with paper. Each cubic centimeter NH_4OH = 0.8cc standard HCl. Each standard cubic centimeter HCl=0.00715 ; HCl=0.0147 $H_2C_4H_4O_6$; 6.0 × 0.8=4.8 standard HCl. 4.8 × 0.0147 = .07056 equivalent of $H_2C_4H_4O_6$ in 10 cc. wine. 1cc. = 0.007056, in terms of percentage=0.706 per cent tartaric acid.

Angelica.—Specific gravity of wine taken by picnometer at temperature of laboratory. Sp. gr. of distillate, picnometer at 15.5. Wine = 1.039; distillate = 0.9768.

Total residue: Evaporated weighed quantity (10.22 grams) in platinum dish over

water bath; dried in oven at 100 degrees for two and one-half hours. Weight was reduced from 17.27 per cent to 16.50 per cent by heating in oven.

Alcohol: Distilled 103.1730 grams wine with 50 cc. water. Distillate weighed 97.372 by volume 19.28, by weight 15.65. As the distillate was 0.9436 of amount taken, then grams. Cooled to 15.5 sp. gr. = 0.9768; found alcohol equivalent in table to be alcohol by volume = $19.28 \times 0.9436 = 18.20$; alcohol by weight = $15.65 \times 0.9436 = 14.77$.

Total acid: Titrated 10 cc. wine against standard NH_4OH. Determined neutral point with litmus paper. Each cc. $NH_4OH = 0.8$ cc. standard HCl; each cubic centimeter standard HCl = 0.00715 HCl = 0.0147 $H_2C_4H_4O_6$.

$6.0 \times .8 = 4.8$, equivalent cc. of standard HCl.

$4.8 \times .0147 = 0.07056$, equivalent of $H_2C_4H_4O_6$ in 10 cc. wine; 1 cc. = 0.007056 in terms of percentage, 0.706 tartaric acid.

MICHIGAN.

The estimates are all made at temperature of 23° and without corrections for temperature.

Angelica.

Specific gravity by Westphal balance	1.039
Specific gravity by picnometer	1.040

Evaporated 10.4 grams (10 cc.) on water bath and dried to a constant weight in air bath, 100° (2 hours); residue, 1.743 grams.

Per cent of solids in wine.

Distilled 104 grams (100 cc.) + 50 cc. water to 100 cc. distillate.

Specific gravity of distillate by picnometer	0.9714
Alcohol, per cent by volume, in wine	24.53
Residue from distillate made up to 100 cc. by water, specific gravity	106.59

All estimates made at 23°.

Claret.

Specific gravity by Westphal balance	.9920
Specific gravity by picnometer	.9925
Solids by evaporating 50 cc. grams	1.55
Per cent solids	3.10

Distilled 100 cc. + 50 cc. water to 100 cc. distillate.

Specific gravity distillate	0.98088
Per cent of alcohol in wine, by volume	15.25
Residue of distillation made up to 100 cc. with water, specific gravity	1.0100

All determinations made at 23°.

NORTH CAROLINA.

Alcohol.—Approximately 100 cc. and 50 cc. of each wine were weighed, diluted in each case to 150 cc. and distilled slowly from a small glass flask of about 250 cc. capacity. A Liebig's condenser was used for condensing the distillate, which latter was collected in a wide-mouth 100 to 110 cc. flask, fitting tight onto the condenser tube by means of a two-hole rubber stopper, one hole for delivery tube of condenser and the other for a safety tube. The distillate flask was left well covered with cold water. The distillate was transferred to the weighing flask at the end of the process and weighed, and the specific gravity taken either by specific-gravity bottle

or Westphal balance, or both. The sp. gr. by the balance was taken at 15.5°; by the bottle at 20°, at which temperature its capacity had been determined.

Extract.—Total solids: Forty to 50 grams of the dry wine and 20 to 30 grams of the sweet wine were weighed from a weighing bottle and evaporated in a platinum dish of about 100 cc. capacity and dried in air bath at 100° for two and one-half hours. The residue from the sweet wine, after cooling and weighing, was again dried two and one-half hours and weighed. Results are given for both.

Extract was also determined by making up the residue from the 100 cc. distillation in case of the claret to original volume and taking the specific gravity, and in case of the sweet wine by evaporating 100 cc. to a paste and bringing to same volume and taking the specific gravity.

Distillation residue was made up to original volume and sp. gr. determined.

	Sp. gr.	Extract.	
		Per cent.	*Average.*
Claret, by bottle	1.0112	2.89	.....
Claret, by Westphal balance	1.0113	2.92	2.91
Angelica, by Westphal balance	1.0675	16.86	16.86

Extract by drying.

	Per cent.
Residue from claret dried 2.5 hours	2.78
Another trial	2.91
Average	2.84
Residue from angelica dried 2.5 hours	15.46
Another trial	16.13
Average	15.79
Same but dried 5 hours	15.00
Another trial	15.25
Average	15.12

Specific gravity of wine.

Of claret taken by bottle	.9945
Of claret taken by Westphal balance	.9943
Of angelica taken by bottle	1.0426
Of angelica taken by Westphal balance	1.0429

EXTRACT.—*Alcohol determinations.*

Wine.	Weight wine taken.	Weight distillate.	Alcohol in distillate by weight.	Alcohol in distillate by volume.	Alcohol in wine by weight.	Alcohol in wine by volume.
	Grams.	*Grams.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Claret	99.0419	102.1897	10.20	12.65	10.52	13.05
	49.5696	101.8220	4.96	6.20	10.19	12.65
Angelica	103.7655	102.8070	15.88	19.55	15.73	19.35
	51.8949	103.2380	7.84	9,75	15.59	19.20

INDIANA.

I believe that the methods used are indicated in the report. When not otherwise stated the methods of Bulletin 31 were followed to the letter. I think closer work on specific gravities could have been made if the work had been conducted in cooler weather. I did not push the drying beyond two and a half hours, although I presume more volatile matter would have gone off if the time had been extended.

All specific gravities were made with flask and with temperature at 15.5°.

Claret.

Specific gravity of distillate at 20° C. (68 F.)..... 0.98230

	Per cent.
.98230 by Table III.....	11.23
Cor. by Table IV	.81
.98334 sp. gr. at 60° F. = ...	10.42 by weight on the distillate.

The specific gravity of wine at 25° C. (77° F.) was .99335
Corrected by Tables II and I= ..... .99518 at 60° F,

Corrections made as follows:

.99335 + .00665 =.....	1.0000 sp. gr. Balling.	
	1.0000 sp. gr. = .0001	
Cor. for temperature.....	.48	.48
		.48

= by Table II, 1.00183
1.00183 — .00665 = .99518.

Therefore 10.42 × .98334 ÷ .99518 = 10.30 per cent by weight on the wine.
By volume:

	Per cent.
.98230 by Table III =.....	13.90
Cor. by Table IV	.90
	13.00 by volume on the distillate.

But the wine was measured at 25° C. (77 F.), therefore 13.00 × .99518 ÷ .99335 = 13.03 per cent by volume on the wine.

Calculations and results on alcohol and specific gravity of wines—corrected.

Specific gravity of distillate at 18° C. (64.4° F.)..... .97530

	Per cent.
.97530 by Table III =.....	16.90
Cor. by Table IV?	.61
.97604 specific gravity at 60° F. = ...	16.29 by weight in distillate.

The specific gravity of the wine at 18 °C.(64.4° F.) was 1.0381, corrected by Table II and I = 1.03877 at 60° F. Then 16.29 × .97604 ÷ 1.03877 = 15.30 per cent by weight on the wine.
By volume:

	Per cent.
.99530 × Table III = ...	20.77
Cor. by Table IV = ...	.77
	20.00 by volume on the distillate.

But the wine was measured at 18° C. (64.4° F.); therefore 20.00 × 1.03877 ÷ 1.0381 = 20.01 per cent by volume on the wine.

Let me ask you a question: What is meant by the words, "by volume" in the heading of Table III (pp. 218–222)? Also, do not the directions in that paragraph (5, p. 224) give percentages by weight instead of by volume? According to my notions the percentage by volume in the distillate is correct for the wine provided that 100 cc. of wine and distillate are measured at standard temperature. Also, do you think the Balling sugar scale is ever reasonably reliable for use in correcting specific gravity in a wine for temperature? I should question it, especially when the wine is in large proportion.

SOUTH CAROLINA.

Determination of alcohol.—One hundred cc. delivered by pipette with a glass-stoppered 100-cc. flask and weighed. Transferred to a 300-cc. distilling flask, washed out weighing flask with 50 cc. distilled water and added washings to distilling flask. Distilled carefully until distillate measured 100 cc. The distillate was weighed and its specific gravity taken at 15.5° C. The per cent of alcohol by weight was taken then form table in Bulletin 31, and thence the per cent of alcohol by weight in the wine calculated.

The determination of alcohol in each wine was also made by determining total acidity and then neutralizing the wine with $\frac{N}{5}$ NaOH sol. before distilling. In this case the wine was weighed out, transferred to distilling flask, using the $\frac{N}{5}$ NaOH sol. diluted to 50 cc. with water, to wash out the weighing flask.

The results obtained were as follows:

Absolute alcohol by weight:

Claret	10.91
Angelica	15.71

Determination of extract.—In case of the claret 50 cc. weighed in a glass-stoppered flask, transferred to a platinum dish, the flask washed, and washings added to the dish. Contents evaporated in water bath until no further diminution of volume was observed, when the dish was transferred to an air bath and the residue heated for two and a half hours at 100°. The result was that given in the foregoing table, 2.99 per cent; 25 cc. weighed and diluted to 50 cc. with water gave 2.93 per cent; 25 cc. weighed and mixed with purified and ignited sand gave 2.82 per cent.

It should be remarked that these determinations were made in the order given on three consecutive days.

In case of the angelica:

	Per cent.
10 cc. weighed, diluted to 50 cc., with water, gave	15.66
10 cc. weighed, diluted to 50 cc., and mixed with sand gave	15.73

Residue from distillation, made up to 100 cc. and tested with Brix spindle, gave, in case of—

Claret, corrected for temperature	2.99
Angelica, corrected for temperature	15.63

DELAWARE.

All estimations rest on weights, not volumes.

Specific gravities.—By the Westphal balance and the specific-gravity flask, the two methods agreeing excellently. I omitted to take the specific gravity of the claret till the sample was used up.

Alcohol.—A weighed quantity (about 100 grams) was distilled with 50 cc. water and several pieces of pumice stone. No fixed alkali was added to fix the volatile acid. The distillate was about 100 cc. It was weighed and its specific gravity was taken, the result being recalculated to 15.5°.

Free acid.—Total free acid was estimated by means of $Ba(OH)_2$ with litmus paper. The volatile acid was not determined in a special portion (with current of steam, etc.), but the alcohol distillates were titrated with $Ba(OH)_2$.

Extract.—A determination was made by drying two and one-half hours at 100° and weighing as per official method. Other estimations were made, as you suggest, by driving off the alcohol and then making up to 100 cc. and taking the specific gravity, the result being recalculated, according to correction tables for Balling's scale, to 17°.

In the case of the sweet wine the results seem to agree, but in the case of the claret they do not.

CALIFORNIA.

	Association method.			Viticultural lab. method.		
	Claret.*	Angelica.*	Angelica.†	Claret.‡	Angelica.‡	Angelica.§
1. Sp. gr. of wine, 15.5	.9944	1.0410		.9938	1.0425	
2. Sp. gr. of de-alcoh. wine, 17½				1.0110	1.0678	
3. Sp. gr. of distillate, 15.5	.9831	.97512		.9832	.9750	
4. Alcoholometer read'g, 15.5				13.20	21.20	
5. Alcohol, weight per ct., 15.5	10.61	17.05	15.98	10.69	17.25	16.13
6. Alcohol, volume per ct., 15.5	13.15	20.95	19.65	13.24	21.19	19.85
7. Solid contents by spindle, from sp. gr. of de-alcoh. wine				2.83	16.50	
8. Extract	3.08	{ 15.63 / 15.68		2.84	16.46	

*By weighing measured quantity, and specific gr's. by picnometer. Tables Bull. 31.
†Corrected by formula. Allen. Vol. I, p. 73, for alcohol in sample.
‡Salleron test, specific gravs. deg. Berlin Spindles and Tables, by Otto Hehner.
§Corrected by formula. Allen, Vol. I, p. 72, for alcohol in sample.

In analysis No. 1 figures under "association" were taken by picnometer at 15.5°; those under "Viticultural" by Berlin spindle. In analysis No. 3 the figures under "association" were obtained by weighing 100 cc. wine at 15.5°, and weighing distillate after distilling to 100 cc. at same temperature and determining sp. gr. with picnometer. To obtain those under "Viticultural" the wine was measured, distilled, made up to same volume and taken at 15.5° with Berlin spindle. In analysis No. 5 "association" alcohol figures were obtained by tables in Bul. 31; "Viticultural," from O. Hehner's tables. In No. 8 under "association" for extract in claret, 50 cc. were weighed, evaporated and placed in air bath at 100° for 2.5 hours and weighed; in angelica 10 cc. were weighed, evaporated, (after III, p. 204, Bul. 31) and heated at 100° in air bath 2.5 hours and weighed. Under "Viticultural" for each wine 2 cc. were weighed, evaporated and dried in air bath at 100° for 2 hours and weighed. Permit me to note, in regard to the determinations of "Extract," that we have followed the practice of weighing up 2 cc., as described in notes, in preference to using larger amounts, as we find we obtain more uniform results, both among themselves and as compared with the specific gravity of the dealcoholized wine converted into solid contents.

ARKANSAS.

Wine weighed in a 100-cc. tared measuring flask at indefinite temperature. Specific gravity taken at 15.5 in a 30 gram picnometer bearing thermometer graduated to $\frac{1}{5}$° C. To insure volume of wine equal to distillate it was in each case computed to exact volume at 15.5°.

Last traces of wine carefully washed into distilling flask with 50 cc. distilled water; distillate collected to mark at indefinite temperature and exact volume calculated as in case of wine. Per cent of alcohol taken from tables. Weight alcohol in distillate divided by the weight of wine taken gives per cent alcohol in wine.

Some difficulty was experienced in taking the specific gravities at the desired temperature, owing to the heated condition of the atmosphere. Much of the time the dew point was above 70° F. The graduated picnometer was each time filled to the mark with the liquid cooled by ice to the proper temperature, and it is believed the precautions taken in weighing were such as to eliminate error.

The total solids were determined as directed and are of course very unsatisfactory for the angelica.

Specific gravity.—Determined in accurately calibrated picnometer, filling at low temperature and allowing to rise to desired point before capping.

Alcohol.—Accurately measured 100 cc. into distilling flask, added 50 cc. water, and distilled 100 cc., receiving distillate at 15°. Specific gravity of distillate determined and alcohol found by tables pp. 218–222, Bulletin 31.

Extract.—Weighed 10 grams wine in platinum dish and dried at boiling point of water in air bath.

Ash.—Burnt residue from extract determination.

Reducing sugar.—Took 50 cc. angelica to 500 cc. (50 cc. claret to 200 cc.). Brought 50 cc. Allihn's solution to boil, added definite volume of wine solutions, boiled two minutes, cooling suddenly by addition of equal volume of cold water. Filtered the oxid upon fine asbestos film in Gooch crucible, and washed away the sulphate with hot water. The sulphate-free oxid was then dissolved in dilute HNO_3(1:3), a little H_2SO_4 added and the HNO_3 driven off by evaporation. The solution, brought to a small bulk, was transferred to an ignited and weighed platinum dish and the copper deposited electrolytically, regulating current by Classen's sulphate of zinc tube. After complete deposition the Cu is washed by decantation, drawing out with a pipette while adding water from a beaker, so that the electrode retains contact with the liquid and prevents solvent action of the H_2SO_4 present.

After complete washing by decantation the current is broken, dish removed and plunged into clean water until absolutely free from H_2SO_4. The water is taken up by several portions of alcohol (90 per cent), and the alcohol-wetted dish finally set in an uncovered desiccator and the alcohol ignited. The residue dries without oxidation. Factor used, 1 part Cu= .5805 parts reducing sugar. (Fuelling's modification.)

Polarizations.—To 100 cc. wine added sufficient basic lead acetate, made to 100 cc. and filtered. Polarization of filtrate calculated to normal dilution. Fifty cc. filtrate is heated for ten minutes at 68° with 5 cc. HCl, cooled quickly to temperature of direct reading, volume made to 55 cc. and reading taken.

Polarization brought to normal dilution.

Some analysts made other determinations besides those requested by the reporter, and their figures are given in the following table:

Angelica.

Sp. gr. at 15.5°.	Total free acid as tartaric.	Volatile free acid as acetic.	Reducing sugar.	Polarization, 26.048: 100.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1.0394	.....	0.03	.....	.....
1.0390	0.500	.....	.....	.....
1.0420	.....	.....	.....	.....
1.0400	.....	.....	.....	.....
1.0410	.....	.....	.....	.....
1.0388	.....	.....	.....	.....
1.0440	.....	.....	.....	.....
1.0426	.....	.....	.....	.....
1.0415	.....	.....	.....	.....
1.0433	.....	.....	16.07	4.38
1.0450	.....	.....	.....	.....
1.0415	.....	.....	.....	.....

Claret.

Sp. gr. at 15.5°.	Total free acid as tartaric.	Volatile free acid as acetic.	Reducing sugar.	Polarization. 26.048 : 100.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
.....	.730	0.08	.....	.....
.9936	.706	.....	.....	.....
.9939	.....	.....	.....	.....
.9925	.....	.....	.....	.....
.9944	.....	.....	.....	.....
.9942	.....	.....	.....	.....
.9953	.....	.....	.....	.....
.9945	.....	.....	.....	.....
.9950	.....	.....	.....	.....
.9949	.....	.....	0.24	0.66
.9950	.....	.....	.....	.....
.9949	.....	.....	.....	.....
.9944	.....	.....	.....	.....

Analyst.

Delaware ..... Penny.
Arizona ..... Collingwood.
South Carolina ..... Shiver.
Michigan ..... Kedzie.
California ..... Colby.
Iowa ..... Eaton.
Indiana ..... Huston.
North Carolina ..... Kilgore.
Arkansas ..... Teller.
United States Department of Agriculture ..... Carr.
Internal Revenue ..... Knorr.
Do ..... Crampton.

Total solids were determined by six analysts by driving off the alcohol from a given volume of the wine, making up to the same volume with water, and then taking the specific gravity of the liquid either with a picnometer or a Balling spindle.

A comparison of these results with those obtained by drying according to the association method are given below.

Comparison of total solids by direct weighing and indirect method.

	Claret.		Angelica.	
	Direct.	Indirect.	Direct.	Indirect.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Penny	3.36	2.87	17.44	17.36
Shiver	2.99	2.99	15.66	15.63
Colby	3.08	2.83	15.65	16.50
Huston	3.22	2.97	16.52	16.89
Kilgore	2.84	2.90	15.79	16.86
Crampton	3.10	2.95	16.81	16.38
Average	3.09	2.92	16.31	16.60

The average shows a result for this method 0.17 per cent lower in the claret and 0.29 per cent higher in the angelica than the direct method. Some authorities claim that the indirect method gives more accurate results than the direct, especially in the case of sweet wines, on account of the difficulty of drying the residue from the latter. It is the method used by the United States gaugers in their work on sweet wines in the field, and I have a large number of results obtained by them checked by direct determinations. I have not yet tabulated or averaged them, but can say that the general agreement is very good in view of the fact that the field work was done by men inexperienced in chemical manipulations, except so far as the reading of a spindle is concerned.

From the notes and descriptions of procedures used by the different analysts it will be seen that several did not rigidly adhere to the prescribed method for the determination of total solids, some using less than the prescribed amount (50 grams) of the dry wine and some prolonging the drying of the residue beyond the prescribed time. The determination of solid matter, or, inversely, of moisture, presents the same difficulty in wines that it does in other substances with which the association has had to deal, enhanced somewhat in the case of sweet wines by the large percentage of moisture-holding matter present.

I must confess that in the work of the laboratory of internal revenue the method of the association is not used. In the approximate determinations made by gaugers Balling's spindle is used as already stated; in fact it is prescribed in the law we execute. But in the exact determinations made in the laboratory the following procedure is in practice: About 5 grams of dry wine or 2 grams of sweet wine are weighed out from a stoppered weighing flask into a platinum dish, placed directly in a drying oven, and dried for three hours at 100°.

I find that the practice of the California laboratory, which handles more samples of wine, probably, than all the rest of the laboratories in the country, is similar, as shown by the following extract from Mr. Colby's letter:

"Permit me to note in regard to the determination of the 'extract' that we have followed the practice of weighing up 2cc. as described in notes in preference to using larger amounts, as we find we obtain more uniform results both among themselves and as compared with the specific gravity of the dealcoholized wine converted into solid contents."

I regret that I have not more comparative determinations by this method which may be termed, for lack of a better name, the method of film drying. The few determinations I have are as follows:

Comparison of total solids by association method and by method of "film drying."

	Claret.		Angelica.	
	Association method.	Film-drying method.	Association method.	Film-drying method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Colby	3.08	2.84	15.65	16.50
Knorr	3.28	2.44	17.38	16.94
Crampton	3.10	3.08	16.81	16.78
Average	3.15	2.79	16.61	16.74

The average shows a minus difference of 0.36 per cent in the case of the claret and a plus difference of 0.13 for the film-drying method as compared with the association method. The results are too few to base any recommendations on, but the method should be investigated further another year.

One analyst reported comparative results on alcohol obtained by neutralizing the

wines with standard alkali solution, showing a slightly higher percentage with the claret and a slightly lower with the angelica.

Similar comparisons were made by Mr. Knorr, who found practically no difference in the results obtained from the wine with and without neutralization.

It will be noticed that I have introduced no figures for the percentage of alcohol by volume in the tables, though in my letter of instructions I particularly requested analysts to return figures for both weight and volume. You will notice also in their reports the difficulties they encountered in making the calculation by volume.

It is without doubt a fact, of which we have only lately become fully cognizant in the Internal-Revenue Office, that alcohol by volume can not be correctly determined in sweet wines, at least with our present tables. This arises from the fact, which is not noticed in any of the books, so far as I am aware, that mixtures of alcohol and water containing in addition a considerable percentage of solid matter have quite a different rate of expansion from mixtures of the same amount of alcohol and water alone.

Ordinarily it might be supposed that such a mixture, a sweet wine for example, being measured out in a definite volume, diluted, and the same volume distilled off at the same temperature, the distillate would contain all the alcohol that was in the original, and its specific gravity referred to a table would indicate the percentage of alcohol by volume in the original. This is the ordinary commercial method. But on comparing results obtained in this way with those obtained by accurate weighing and conversion of weights into volumes, or by getting percentage of alcohol by weight and converting this into percentage by volume by using the specific gravity figure of absolute alcohol, it was found that the figures were uniformly and invariably lower, even to the extent of 2 or 3 per cent.

The only explanation possible is that the alcohol found in the distillate by the table occupies a smaller volume in the wine on account of the presence of the solids than it does in the distillate where it is mixed with water alone, the tables used having been calculated for the latter condition only.

You will understand the difficulty we of the internal-revenue office labor under on account of this matter when I tell you that the sweet-wine clause of the McKinley bill, under which we operate, states the legal limitations for the fortification of such wines in terms of alcohol by volume only. I think you will agree with me, however, in my statement, that we have at present no way of arriving accurately at the percentage of alcohol by volume in sweet wines.

RECOMMENDATIONS.

In the matter of recommendations I have the following to offer:

(1) That the following procedure for the determination of alcohol, giving more explicit and detailed directions than the present method, be substituted for the latter.

Weigh about 100 grams of wine and distill in a proper distilling apparatus (provided with a glass condenser and mercury valve), after the addition of 50 cc. of water. When about 100 cc. of distillate have come over, the operation is stopped and the distillate weighed. Its specific gravity at 15.5° is then ascertained, by means either of a picnometer or a Westphal balance controlled by a picnometer, and the corresponding per cent of alcohol by weight obtained from the official tables. This figure multiplied by the weight of distillate and the result divided by the weight of sample taken gives the per cent of alcohol by weight contained in the sample.

(2) Under the head of "Extract," after "on a water bath," insert "to a sirupy consistency."

(3) Instead of the sentence beginning "In sweet wines, etc.," substitute the following:

"In the case of sweet wines, that is, those containing more than 4 per cent of solid matter, not more than 10 cc. (weighed) should be taken."

(4) As a substitute method for total solids in sweet wines, the following:

"Measure out carefully a definite volume of wine, transfer to an evaporating dish, and evaporate on a water bath until its volume is reduced to about one-third of its original bulk. Transfer the dealcoholized liquid to the graduated vessel in which it was originally measured out, rinse the dish carefully, bring it to the same temperature as when originally measured, and make it up to the same volume, mix thoroughly, and ascertain the specific gravity of the liquid, either by means of a picnometer or by an accurately standardized Balling or Brix spindle. The per cent of the total solids, in case the picnometer is used, may be taken from the official tables herewith."

The remaining determinations may be allowed to stand as they are, although I think the determinations under the head of "Abnormal constituents of wines" may properly be done away with.

I would also recommend that the "method used for the approximate determination of the alcohol," etc., beginning on page 208, be stricken out. It was never intended that this procedure should be incorporated into the association methods last year; only that it be given a place in the body of the bulletin. I would recommend the retention of the table for alcohol, however (Table III), and that its heading be changed to read "Percentage of alcohol by weight and by volume." Also Table I for the correction of the readings of Balling spindles for different temperatures. Table No. II should be cut out. The analyst made the mistake this year of using it to correct his specific gravities.

I would suggest in the work for next year the further investigation of the method of film drying for the estimation of total solids; also the question of the proper calculation and expression of percentage of alcohol by volume in sweet wines.

Mr. KEDZIE. I move that it be adopted.

Seconded and adopted.

Mr. CRAMPTON read his first recommendation.

The second recommendation was read by the reporter.

Mr. SCOVELL. In order to expedite business, I move that each recommendation of the reporter be considered approved as read if there be no objections.

Adopted.

The PRESIDENT. So that the second recommendation is adopted unless there is an objection.

The third recommendation was read by the reporter.

The PRESIDENT. If there is no objection this also will be adopted.

The fourth recommendation was read by the reporter.

The PRESIDENT. Are there any objections to adopting this recommendation?

There being none the recommendation was adopted.

Mr. CRAMPTON. I think the determinations under the head of "Abnormal constituents of wines" (page 207) should be stricken out.

The PRESIDENT. Do you make that as a recommendation?

Mr. CRAMPTON. Yes, sir.

The PRESIDENT. There is no objection and it is adopted.

Mr. CRAMPTON. I recommend that the method used for the approximate determination of the alcohol and sugar contents of sweet wines by United States Internal Revenue officers, (page 208) be stricken out.

It was not intended that this should be incorporated in the association methods last year. I would recommend the retention of Table I and Table III, and that Table II be cut out.

The PRESIDENT. There seems to be no objection to these recommendations and they will stand adopted.

Is there any other business to come before the meeting? I have to announce to the association that I shall take occasion within a very short time to appoint the reporters for the ensuing year, and that they will be notified immediately after the appointment is made of their duties.

Mr. WILEY. And the abstract committee?

The PRESIDENT. Was that finally passed upon?

Mr. WILEY. Yes.

The PRESIDENT. Well, that committee will also be considered.

Mr. WOODS. There is one thing that I think is necessary to all who come to these meetings, and that is that we get acquainted with one another as much as possible. A year ago we met in connection with the American Association at about the same time. This year no hotels were recommended as headquarters, and it seems to me important that we have some hotel for chemists' headquarters, so that we can all come together, at least as many as can, and, therefore, I move that the secretary be instructed to recommend some hotel which shall be used as headquarters for the meeting, wherever it shall be held.

Adopted.

Mr. WOODS. I move that we adjourn to assemble at the call of the executive committee.

Adopted.

Adjourned *sine die*.

NOTE.—In spite of the repeated requests made that the reporters send in their manuscripts in shape to be given directly to the printer, the secretary regrets to say that in at least three instances reports were in such shape as to render correct editing almost impossible. Any errors occurring therefore must be attributed to the original manuscript.

LIST OF OFFICIAL CHEMISTS.

CHEMISTS CONNECTED WITH THE STATE AGRICULTURAL EXPERIMENT STATIONS.

[NOTE BY THE SECRETARY.—No complete list of official chemists, such as was called for by the association, is in existence. The inserted list of chemists connected with agricultural colleges and with State experiment stations is taken from the Organization List of the Agricultural Experiment Stations and Agricultural Schools and Colleges in the United States, published by the Department of Agriculture, June, 1892.]

State.	Location.	Chemist.
Alabama	Auburn	N. T. Lupton.
		J. T. Anderson.
		L. W. Wilkinson.
		R. E. Noble.
		C. L. Hare.
Arizona	Tucson	C. B. Collingwood.
		J. A. Heberly.
Arkansas	Fayetteville	G. L. Teller.
		F. W. Killen.
California	Berkeley	E. W. Hilgard.
		G. E. Colby.
		R. H. Loughridge.
Colorado	Fort Collins	D. O'Brine.
Connecticut (State)	New Haven	E. H. Jenkins.
		A. L. Winton, jr.
		T. B. Osborne.
		A. W. Ogden.
		C. L. Voorhees.
Connecticut (Storrs)	Storrs	C. D. Woods.
		H. M. Smith.
Delaware	Newark	C. L. Penny.
Florida	Lake City	J. M. Pickell.
		J. J. Earle.
Georgia	Experiment	R. E. Hardee.
		H. C. White.
Illinois	Champaign	E. H. Farrington.
		E. K. Nelson.
Indiana	Lafayette	H. A. Huston.
Iowa	Ames	G. E. Patrick.
		E. N. Eaton.
Kansas	Manhattan	G. H. Failyer.
		J. T. Willard.
Kentucky	Lexington	M. A. Scovell.
		A. M. Peter.
		H. E. Curtis.

State.	Location.	Chemist.
Louisiana (sugar)	Audubon Park, New Orleans	W. C. Stubbs.
		J. T. Crawley.
Louisiana (State)	Baton Rouge	B. B. Ross.
		R. E. Blouin.
Louisiana (north)	Calhoun	Maurice Bird.
Maine	Orono	W. H. Jordan.
		J. M. Bartlett.
		L. H. Merrill.
Maryland	College Park	H. J. Patterson.
Massachusetts (State)	Amherst	C. A. Goessmann.
		C. S. Crocker.
		R. B. Moore.
		H. D. Haskins.
		C. H. Jones.
		C. H. Johnson.
Michigan	Agricultural College	R. C. Kedzie.
		W. L. Rossman.
		H. E. Harrison.
Minnesota	St. Anthony Park	H. Snyder.
Mississippi	Agricultural College	W. L. Hutchinson.
		L. G. Patterson.
Missouri	Columbia	P. Schweitzer.
		C. P. Fox.
Nebraska	Lincoln	H. H. Nicholson.
		Rachel Lloyd.
		T. L. Lyon.
Nevada	Reno	N. E. Wilson.
New Hampshire	Hanover	F. W. Morse.
		C. L. Parsons.
		E. P. Stone.
New Jersey (State)	New Brunswick	E. B. Voorhees.
		L. A. Voorhees.
		J. P. Street.
New Jersey (college)	New Brunswick	P. T. Austen.
		C. S. Cathcart.
New Mexico	Las Cruces	E. O. Wooton.
		Arthur Goss.
New York (State)	Geneva	L. L. Van Slyke.
		Peter Collier.
		W. P. Wheeler.
		C. G. Jenter.
		A. L. Knisely.
		Shinichi Ando.
		W. H. Andrews.
		B. L. Murray.
New York (Cornell)	Ithaca	G. C. Caldwell.
		G. W. Cavanaugh.
North Carolina	Raleigh	H. B. Battle.
		B. W. Kilgore.
		F. B. Carpenter.
		T. L. Blalock.
		J. S. Meng.
North Dakota	Fargo	E. F. Ladd.
		W. H. Whalen.
Ohio		F. J. Falkenbach.

State.	Location.	Chemist.
Oklahoma	Stillwater	G. L. Holter.
Oregon	Corvallis	G. W. Shaw.
		Dumont Lotz.
Pennsylvania	State College	William Frear.
		E. J. Haley.
		W. S. Sweetser.
		J. A. Fries.
		J. W. Fields.
Rhode Island	Kingston	H. J. Wheeler.
		B. L. Hartwell.
South Carolina	Fort Hill	M. B. Hardin.
		C. W. Sims.
		F. S. Shiver.
South Dakota	Brookings	J. H. Shepard.
		C. G. Hopkins.
Tennessee	Knoxville	C. W. Dabney.
		J. B. McBryde.
Texas	College Station	H. H. Harrington.
		D. Adriance.
		P. S. Tilson.
Utah	Logan	W. P. Cutter.
Vermont	Burlington	J. L. Hills.
		B. O. White.
Virginia	Blacksburg	R. J. Davidson.
		T. L. Watson.
Washington	Pullman	G. G. Hitchcock.
West Virginia	Morgantown	R. J. J. De Roode.
Wisconsin	Madison	S. M. Babcock.
		F. W. Woll.
Wyoming	Laramie	J. D. Conley.
		E. E. Slosson.

CHEMISTS CONNECTED WITH AGRICULTURAL COLLEGES.

[Those marked * occur in the preceding list.]

Anderson, Joseph. Rodney, Miss. Alcorn Agricultural and Mechanical College.
Appleton, J. H. Providence, R. I. Brown University.
Aubert, Alfred B. Orono, Me. Maine State College of Agriculture and Mechanic Arts.
*Babcock, S. M. Madison, Wis. College of Agriculture of the University of Wisconsin.
Bennett, A. A. Ames, Iowa. Iowa State College of Agriculture and Mechanic Arts.
Bentley, W. B. Fayetteville, Ark. Arkansas Industrial University.
Bloomfield, L. M. Columbus, Ohio. Ohio State University.
Breese, C. M. Manhattan, Kans. Kansas State Agricultural College.
*Caldwell, G. C. Ithaca, N. Y. College of Agriculture of Cornell University.
Chamberlain, J. Ames, Iowa. Iowa State College of Agriculture and Mechanic Arts.
Chester, A. H. New Brunswick, N. J. Rutgers Scientific School.
Colby, D. W. Orono, Me. Maine State College of Agriculture and the Mechanic Arts.
*Collingwood, C. B. Tucson, Ariz. College of Agriculture of the University of Arizona.

* Cutter, W. P. Logan, Utah. Agricultural College of Utah.
* Dabney, C. W. Knoxville, Tenn. State Agricultural and Mechanical College of the University of Tennessee.
Dye, C. A. Columbus, Ohio. Ohio State University.
* Failyer, G. H. Manhattan, Kans. Kansas State Agricultural College.
* Frear, William. State College, Pa. Pennsylvania State College.
Fulmer, H. E. Lincoln, Nebr. Industrial College of the University of Nebraska.
* Goessmann, C. A. Amherst, Mass. Massachusetts Agricultural College.
* Goss, Arthur. Las Cruces, N. Mex. New Mexico College of Agriculture and Mechanic Arts.
* Harrington, H. H. College Station, Tex. State Agricultural and Mechanical College of Texas.
* Hilgard, E. W. Berkeley, Cal. College of Agriculture of the University of California.
Hird, J. D. College Park, Md. Maryland Agricultural College.
* Hitchcock, G. G. Pullman, Wash. Washington Agricultural College and School of Science.
* Holter, G. L. Stillwater, Okla. Agricultural and Mechanical College of Oklahoma.
* Huston, H. A. Lafayette, Ind. School of Agriculture, Horticulture, and Veterinary Science of Purdue University.
* Hutchinson, W. L. Agricultural College, Miss. Agricultural and Mechanical College of Mississippi.
Johnson, S. W. New Haven, Conn. Sheffield Scientific School of Yale University.
Kastle, J. H. Lexington, Ky. Agricultural and Mechanical College of Kentucky.
Kedzie, F. S. Agricultural College, Mich. Michigan Agricultural College.
* Kedzie, R. C. Agricultural College, Mich. Michigan Agricultural College.
Keffer, Fred. Columbus, Ohio. Ohio State University.
* Ladd, E. F. Fargo, N. Dak. North Dakota Agricultural College.
* Lloyd, Rachel. Lincoln, Nebr. Industrial College of the University of Nebraska.
* Lotz, D. Corvallis, Oregon. Oregon State Agricultural College.
* Loughbridge, R. H. Berkeley, Cal. College of Agriculture of the University of California.
* Lupton, N. T. Auburn, Ala. Agricultural and Mechanical College, Alabama Polytechnic Institute.
Lynes, J. C. Milledgeville, Ga. Middle Georgia Military and Agricultural College.
* Lyon, T. L. Lincoln, Nebr. Industrial College of the University of Nebraska.
Menke, A. E. Fayetteville, Ark. Arkansas Industrial University.
Merrill, N. F. Burlington, Vt. University of Vermont and State Agricultural College.
Mitchell, G. A. New Brunswick, N. J. Rutgers Scientific School.
Mixter, W. G. New Haven, Conn. Sheffield Scientific School of Yale University.
* Morse, F. W. Hanover, N. H. New Hampshire College of Agriculture and Mechanic Arts.
McBryde, J. M. Blacksburg, Va. Virginia Agricultural and Mechanical College.
McDonnell, H. B. College Park, Md. Maryland Agricultural College.
* Nicholson, H. H. Lincoln, Nebr. Industrial College of the University of Nebraska.
Norton, S. A. Columbus, Ohio. Ohio State University.
* O'Brine, D. Fort Collins, Colo. The State Agricultural College of Colorado.
Palmer, A. W. Champaign, Ill. College of Agriculture of the University of Illinois.
Parr, S. W. Champaign, Ill. College of Agriculture of the University of Illinois.
* Parsons, C. L. Hanover, N. H. New Hampshire College of Agriculture and Mechanic Arts.
* Patrick, G. E. Ames, Iowa. Iowa State College of Agriculture and Mechanic Arts.
Peebles, A. B. Mansfield, Conn. Storrs Agricultural School.

Perkins, W. R. Agricultural College, Miss. Agricultural Mechanical College of Mississippi.
Peter, Robert. Lexington, Ky. Agricultural and Mechanical College of Kentucky.
Phillips, J. Warne. Reno, Nev. School of Agriculture of the Nevada State University.
* Pickell, J. M. Lake City, Fla. Florida State Agricultural and Mechanical College.
Pond, G. G. State College, Pa. Pennsylvania State College.
Price, R. C. Blacksburg, Va. Virginia Agricultural and Mechanical College.
Rising, W. B. Berkeley, Cal. College of Agriculture of the University of California.
* Ross, B. B. Baton Rouge, La. Louisiana State University and Agricultural and Mechanical College.
Roy, V. L. New Orleans, La. Southern University and Agricultural and Mechanical College.
* Schweitzer, P. Columbia, Mo. College of Agriculture and Mechanic Arts of the University of Missouri.
* Shaw, G. W. Corvallis, Oreg. Oregon State Agricultural College.
* Shepard, J. H. Brooking, S. Dak. South Dakota Agricultural College.
* Slosson, E. E. Laramie, Wyo. College of Agriculture of the University of Wyoming.
* Snyder, Harry. St. Anthony Park, Minn. College of Agriculture of the University of Minnesota.
Speyers, C. L. New Brunswick, N. J. Rutgers Scientific School.
Storer, F. H. Jamaica Plain, Mass. Bussey Institution of Harvard University.
Thurliman, L. Ames, Iowa. Iowa State College of Agriculture and Mechanic Arts.
Washburn, J. H. Kingston, R. I. Rhode Island College of Agriculture and Mechanic Arts.
Weber, H. A. Columbus, Ohio. Ohio State University.
Wellington, Charles. Amherst, Mass. Massachusetts Agricultural College.
*White, H. C. Athens, Ga. Georgia State College of Agriculture and Mechanic Arts.
Whitehill, A. R. Morgantown, W. Va. West Virginia University.
* Willard, J. T. Manhattan, Kans. Kansas State Agricultural College.
Withers, W. A. Raleigh, N. C. The North Carolina College of Agriculture and Mechanic Arts.
Wolf, T. R. Newark, Del. Delaware College.
* Woll, F. W. Madison, Wis. College of Agriculture of the University of Wisconsin.

CHEMISTS CONNECTED WITH THE U. S. DEPARTMENT OF AGRICULTURE.

Allen, E. W.....Office of Experiment Stations.
Bigelow, W. D .....Division of Chemistry.
Carr, Oma.....Division of Chemistry.
De Schweinitz, E. A .....Bureau of Animal Industry.
Edson, H.....Division of Chemistry.
Emery, J. A.....Bureau of Animal Industry.
Ewell, E. E.....Division of Chemistry,
Krug, William .....Division of Chemistry.
Maxwell, W.....Division of Chemistry.
McElroy, K. P .....Division of Chemistry.

CHEMISTS CONNECTED WITH THE U. S. DEPARTMENT OF AGRICULTURE—Continued.

Packard, R. L	Weather Bureau.
Runyan, E. G	Division of Chemistry.
Spencer, G. L	Division of Chemistry.
Sutton, D. C	Division of Chemistry.
Trescot, T. C	Division of Chemistry.
Wiley, H. W	Division of Chemistry.

TEMPORARILY EMPLOYED.

Fuelling, J. L	Division of Chemistry.
Sanborn, T. F	Division of Chemistry.
Wedderburn, Aug	Division of Chemistry.

CHEMISTS CONNECTED WITH THE U. S. TREASURY DEPARTMENT

Crampton, C. A	Bureau of Internal Revenue.
Knorr, A. E	Bureau of Internal Revenue.

OFFICERS, REPORTERS, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS OF THE UNITED STATES FOR 1892–'93.

PRESIDENT.

Mr. S. M. BABCOCK, chemist of the Wisconsin Experiment Station.

VICE-PRESIDENT.

Mr. E. B. VOORHEES, chemist of the New Jersey Experiment Station.

SECRETARY.

Mr. H. W. WILEY, chief chemist U. S. Department of Agriculture.

EXECUTIVE COMMITTEE.

The president, vice-president, and secretary, *ex officiis*.
Messrs. B. W. KILGORE, of Raleigh, N. C.
H. H. HARRINGTON, of College Station, Tex.

REPORTERS.

Phosphoric acid.—R. J. J. De Roode, Morgantown, W. Va.
Nitrogen.—C. L. Parsons, Hanover, N. H.
Potash.—Norman Robinson, Tallahassee, Fla.
Soils and ash.—E. W. Hilgard, Berkeley, Cal.
Dairy products.—A. L. Winton, New Haven, Conn.
Foods and feeding stuffs low in fiber.—F. W. Woll, Madison, Wis.
Foods and feeding stuffs high in fiber.—G. L. Teller, Fayetteville, Ark.
Fermented liquors.—C. A. Crampton, Washington, D. C.
Sugar.—G. L. Spencer, Washington, D. C.

COMMITTEE TO CONFER WITH WORLD'S FAIR AUXILIARY CONGRESS.

S. M. Babcock, Madison, Wis.
H. W. Wiley, Washington, D. C.
M. A. Scovell, Lexington, Ky.
H. A. Huston, Lafayette, Ind.
L. A. Voorhees, New Brunswick, N. J.

ABSTRACT COMMITTEE.

William Frear, State College, Pa.
A. L. Winton, New Haven, Conn.
E. W. Allen, Washington, D. C.
B. B. Ross, Baton Rouge, La.
F. W. Woll, Madison, Wis.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects shall be (1) to secure uniformity and accuracy of the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members, and to all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary who shall also officiate as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a reporter for each of the subjects to be considered by the association.

It shall be the duty of these reporters to prepare and distribute samples and standard reagents to members of the association and others desiring the same; to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee and announced at least three months before the time of meeting.

(7) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(8) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(9) All proposed alterations or amendments to this constitution shall be referred to a select commitee of three at a regular meeting, and after report from such committee may be adopted by a vote of two-thirds of the members present entitled to vote.

OFFICIAL METHODS OF ANALYSIS ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS AT ITS MEETING, AUGUST 25, 26, 27, 1892.

PREPARATION OF SAMPLE.

The sample should be well intermixed, finely ground, and passed through a sieve having circular perforations 1 mm. in diameter. The process of grinding and sifting should take place as rapidly as possible, so that there should be no loss or gain of moisture during the operation.

DETERMINATION OF MOISTURE.

(*a*) In potash salts, sodium nitrate, and ammonium sulphate, heat from 1 to 5 grams at 130° until the weight is constant, and determine the loss in weight as moisture. (*b*) In all other fertilizers heat 2 grams, or 5 grams if the sample be very coarse, for 5 hours at 100° in a steam bath.

METHOD OF DETERMINING PHOSPHORIC ACID.

Water soluble phosphoric acid.—Place 2 grams in a small filter about 9 cm. in diameter, wash with successive small portions of water, allowing each portion of water to pass through before adding more until the filtrate measures not less than 250 cc. After mixing, take an aliquot portion corresponding to 0.5 grams of the original substance, and determine the phosphoric acid as under total phosphoric acid.

Citrate insoluble phosphoric acid.—Wash the residue in the filter from the above determination into a 200 cc. flask with 100 cc. of strictly neutral ammonium-citrate solution of 1.09 sp. gr., prepared as hereafter directed. Cork the flask securely and place it in a water bath kept at the temperature of 65°. The water bath should be of such a size that the introduction of the flask, or flasks, shall not cause a reduction of the temperature of more than 2°. After the introduction of the flasks the temperature of the bath should be brought as rapidly as possible to 65° and maintained at this point for 30 minutes. The contents of the flask should then be filtered rapidly and washed with water at about the temperature of 65°. With fertilizers containing iron or aluminum the addition of a small amount of citrate solution to the wash water will give clear washings. Transfer the filter and its contents to a crucible, ignite until the organic matter is destroyed, add from 10 to 15 cc. of concentrated hydrochloric acid, and digest over a small flame until the phosphate is dissolved. Dilute the solution to 200 cc. If desired the filter and its contents can be treated according to methods 1, 2, or 3, under total phosphoric acid. Mix well, filter through a dry filter, take a definite portion of the filtrate and determine phosphoric acid according to the method for total phosphoric acid.

In case a determination of citrate insoluble phosphoric acid is required in non-acidulated goods, it is to be made by treating 2 grams of the phosphatic material, without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, etc.), the residue insoluble in ammonium citrate is to be treated by one of the processes described below under "total phosphoric acid," 1, 2, or 3.

Total phosphoric acid.—In case of ignition, the ignited material is to be dissolved in hydrochloric acid. Weigh 2 grams and treat by one of the following meth-

ods: (1) Evaporate with 5 cc. of magnesium nitrate, ignite and dissolve in hydrochloric acid. (2) Boil with 20 to 30 cc. of strong sulphuric acid, adding 2 to 4 grams of sodium or potassium nitrate at the beginning of the digestion and a small quantity after the solution has become nearly colorless, or adding the nitrate in small portions from time to time. A Kjeldahl flask graduated to 250 cc. is recommended. After the solution is colorless add 150 cc. of water and boil for a few minutes, cool, and make up to volume. (3) Treat with sulphuric acid and mercury or mercuric oxid, as in the Kjeldahl method, using a flask graduated to 250 cc. When the digestion is completed add 150 cc. of water, boil for a few minutes, cool, and make up to volume. These methods are to be used in case of fertilizers containing cotton seed or much organic matter. (4) Solution in 30 cc. of concentrated nitric acid with a small quantity of hydrochloric acid. (5) Add 30 cc. of concentrated hydrochloric acid, heat, and add cautiously in small quantities at a time about 0.5 gram of finely pulverized potassium chlorate. (6) Solution in 15 to 30 cc. of strong hydrochloric acid and 5 to 10 cc. of nitric acid. This method is recommended for fertilizers containing much iron or aluminum phosphate. Boil until all phosphates are dissolved and all organic matter is destroyed; cool and dilute to 200 or 250 cc., mix and pass through a dry filter; take an aliquot part of the filtrate corresponding to 0.25 gram, 0.50 gram, or 1 gram; neutralize with ammonia, and clear with a few drops of nitric acid. (In case hydrochloric or sulphuric acid has been used as a solvent, add about 15 grams of dry ammonium nitrate or its equivalent.)

To the hot solutions for every decigram of P_2O_5 that is present add 50 cc. of molybdic solution. Digest at about 65° for one hour, filter and wash with water or ammonium-nitrate solution. Test the filtrate by renewed digestion and addition of more molybdic solution. Dissolve the precipitate on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (about one drop per second), stirring vigorously. After fifteen minutes add 30 cc. of ammonia solution of density 0.95. Let stand for some time; two hours is usually enough. Filter, wash with dilute ammonia, ignite at white heat for ten minutes, and weigh.

Citrate soluble phosphoric acid.—The sum of the water soluble and citrate insoluble subtracted from the total gives the citrate soluble.

PREPARATION OF REAGENTS.

(1) *Ammonium citrate solution.*—(*a*) Mix 370 grams of commercial citric acid with 1,500 cc. of water; nearly neutralize with commercial ammonia; cool; add ammonia until exactly neutral (testing with saturated alcoholic solution of corallin) and bring to volume of 2 liters. Test the specific gravity, which should be 1.09 at 20°, before using.

(*b*) Alternate method. To 370 grams of commercial citric acid add commercial ammonia, sp. gr. 0.96, until nearly neutral; reduce the specific gravity to nearly 1.09 and make exactly neutral, testing as follows: Prepare a solution of fused calcium chlorid 200 grams to the liter, and add four volumes of strong alcohol. Make the mixture exactly neutral, using a small amount of freshly prepared corallin solution as a preliminary indicator, and test finally by withdrawing a portion, diluting with an equal volume of water, and testing with cochineal solution. Fifty cc. of this solution will precipitate the citric acid from 10 cc. of the citrate solution. To 10 cc. of the nearly neutral citrate solution add 50 cc. of the alcoholic calcium chlorid solution, stir well, filter at once through a folded filter, dilute with an equal volume of water, and test the reaction with neutral solution of cochineal.

(2) *Molybdic solution.*—Dissolve 100 grams of molybdic acid in 400 grams, or 417 cc., of ammonia, sp. gr. 0.96, and pour the solution thus obtained into 1,500 grams, or 1,250 cc., of nitric acid, sp. gr. 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° deposits no yellow precipitate of ammonium

phosphomolybdate. Decant the solution from any sediment and preserve in glass-stoppered vessels.

(3) *Ammonium nitrate solution.*—Dissolve 200 grams of commercial ammonium nitrate in water and bring to a volume of 2 liters.

(4) *Magnesia mixture.*—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding an excess of the latter. Add a little calcined magnesia in excess and boil a few minutes to precipitate iron, alumina, and phosphoric acid; filter; add 280 grams of ammonium chlorid, 700 cc. of ammonia of sp. gr. 0.96, and water enough to make a volume of 2 liters. Instead of the solution of 22 grams of calcined magnesia 110 grams of crystallized magnesium chlorid ($MgCl_2$, $6H_2O$) may be used.

(5) *Dilute ammonia for washing.*—One volume of ammonia sp. gr. 0.96 mixed with three volumes of water, or usually one volume of concentrated ammonia with six volumes of water.

(6) *Nitrate of magnesia.*—Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess; boil; filter from the excess of magnesia, ferric oxid, etc., and bring to volume of 2 liters.

METHODS OF DETERMINING POTASH.

METHOD OF LINDO-GLADDING.

(1) *In superphosphates.*—Boil 10 grams with 300 cc. of water 30 minutes. To the hot solution add ammonia in slight excess, and then a sufficient quantity of ammonium oxalate to precipitate all the lime present; cool and make up to 500 cc., mix thoroughly, and filter through a dry filter; take 50 cc., corresponding to 1 gram, evaporate nearly to dryness, add 1 cc. of dilute H_2SO_4 (1 to 1), evaporate to dryness and ignite to whiteness. As all the potash is in form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. This residue is dissolved in hot water, plus a few drops of HCl: and a slight excess of platinum solution is now added. This solution is then evaporated to a thick paste in a small dish, and 80 per cent alcohol added In evaporating, special precaution should be taken to prevent absorption of ammonia. The precipitate is washed thoroughly with alcohol by decantation and on the filter, as usual. The washing should be continued even after the filtrate is colorless. Ten cc. of the NH_4Cl solution, prepared as hereafter directed, are now run through the filter, or the washing may be performed in the dish. The 10 cc. will contain the bulk of the impurities, and are thrown away. Fresh portions of 10 cc. of the NH_4Cl are now run through the filter several times (5 or 6). The filter is then washed thoroughly with pure alcohol, dried, and weighed as usual. Care should be taken that the precipitate is perfectly soluble in water. The platinum solution used contains 1 gram of metallic platinum in every 10 cc. To prepare the washing solution of NH_4Cl, place in a bottle 500 cc. of H_2O, 100 grams of NH_4Cl; shake till dissolved. Now, pulverize 5 or 10 grams of K_2PtCl_6, put in the bottle, and shake at intervals for six or eight hours; let settle over night, then filter off the liquid into a second bottle. The first bottle is then ready for preparation of a fresh supply when needed.

(2) *Muriates of potash.*—In the analysis of these salts an aliquot portion containing 0.5 grams is evaporated with 10 cc. of platinum solution and a few drops of HCl and washed as before.

(3) *Sulphate of potash, kainit, etc.*—In the analysis of kainit, dissolve 10 grams of the pulverized salt in 300 cc. of boiling water, add ammonia to slight excess, then a sufficient quantity of ammonium oxalate to throw down all lime present; cool and make up to 500 cc., mix thoroughly, and filter on a dry filter; take 25 cc., corresponding to 0.5 grams, and proceed to remove the ammonia, as in the analysis of superphosphates; dissolve the residue in hot water plus a few drops of HCl and add 15 cc. of platinum solution. In the analysis of high-grade sulphate and of double-manure

salt (K_2SO_4, $MgSO_4$, containing about 27 per cent of K_2O), make up the solution as above, but omit the precipitation, evaporation, etc.; take an aliquot part equal to 0.5 grams and add 15 cc. of platinum solution. In all cases special care must be taken in the washing with alcohol to remove all the double platinum-sodium chlorid if present. The washing should be continued some time after the filtrate is colorless. Twenty-five cc. of the NH_4Cl solution are employed instead of 10 cc., and the 25 cc. poured through at least 6 times to remove all sulphates and chlorids. Wash finally with alcohol; dry and weigh as usual.

(4) *Organic compounds.*—In case the potash is contained in organic compounds like tobacco stems, cotton-seed hulls, etc., weigh out 10 grams, saturate with strong sulphuric acid, and ignite in a muffle to destroy organic matter. Add a little strong HCl to moisten the mass and warm slightly so as to loosen it in the dish. Proceed then as in (1).

ALTERNATE METHOD FOR POTASH.

Take 10 grams of the prepared sample, boil for 10 minutes with 200 cc. of water, and, after cooling and without filtering, make up to 1,000 cc. and filter through a dry filter. If the sample has 10 to 15 per cent of K_2O (kainit), take 50 cc. of the filtrate; if from 2 to 3 per cent of K_2O (ordinary potash fertilizers), take 100 cc. of the filtrate. In each case make the volume up to 150 cc., heat to 100°, and add, drop by drop with constant stirring, slight excess of barium chlorid, without filtering; in the same manner add barium hydrate in slight excess. Filter while hot and wash until the precipitate is free of chlorids. Add to the filtrate 1 cc. of strong ammonium hydrate, and then a saturated solution of ammonium carbonate until the excess of barium is precipitated. Heat; add now, in fine powder, 0.5 grams pure oxalic acid or 0.75 grams of ammonium oxalate. Filter; wash free of chlorids, evaporate the filtrate to dryness in a platinum dish, and ignite carefully over the free flame below red heat until all volatile matter is driven off.

The residue is now digested with hot water, filtered through a small filter, and washed with successive small portions of water until the filtrate amounts to 30 cc. or more. To this filtrate, after adding 2 drops of strong hydrochloric acid, are added, in a porcelain dish, 5 to 10 cc. of a solution of 10 grams of platinic chlorid in 100 cc. of water. The mixture is now evaporated on a water bath to a thick sirup, as above, treated with 80 per cent alcohol, washed by decantation, collected in a Gooch crucible or other form of filter, washed with strong alcohol, afterwards with 5 cc. of ether, dried for 30 minutes at 100°, and weighed.

It is desirable, if there be an appearance of foreign matter in the double salt, that it should be washed, according to the previous method, with 10 cc. of the half concentrated solution of NH_4Cl, which has been saturated by shaking with K_2PtCl_6.

FACTORS.

The use of the factors 0.3056 for converting K_2PtCl_6 to KCl and 0.19308 for converting to K_2O is continued.

METHODS FOR THE DETERMINATION OF NITROGEN.

THE ABSOLUTE OR CUPRIC OXID METHOD.

[Applicable to all nitrogen determinations.]

The apparatus and reagents needed are as follows:

APPARATUS.

Combustion tube of best hard Bohemian glass, about 66 cm. long and 12.7 mm. internal diameter.

Azotometer of at least 100 cc. capacity, accurately calibrated.

Sprengel mercury air pump.

Small paper scoop, easily made from stiff writing paper.

REAGENTS.

Coarse cupric oxide or wire form.—To be ignited and cooled before using.

Fine cupric oxid.—Prepared by pounding ordinary cupric oxid in a mortar.

Metallic copper.—Granulated copper, or fine copper gauze, reduced and cooled in a current of hydrogen.

Sodium bicarbonate.—Free from organic matter.

Caustic potash solution.—Make a supersaturated solution of caustic potash in hot water. When absorption of carbonic acid ceases to be prompt, the solution must be discarded.

MANIPULATION.

Filling the tube.—Of ordinary commercial fertilizers take 1 to 2 grams for analysis. In the case of highly nitrogenized substances the amount to be taken must be regulated by the amount of nitrogen estimated to be present. Fill the tube as follows: (1) About 5 cm. of coarse cupric oxid. (2) Place on the small paper scoop enough of the fine cupric oxid to fill, after having been mixed with the substance to be analyzed, about 10 cm. of the tube; pour on this the substance, rinsing the watch glass with a little of the fine oxid, and mix thoroughly with a spatula; pour into the tube, rinsing the scoop with a little fine oxid. (3) About 30 cm. of coarse cupric oxid. (4) About 7 cm. of metallic copper. (5) About 6 cm. of coarse cupric oxid (anterior layer). (6) A small plug of asbestos. (7) 0.8 to 1 gram of sodium bicarbonate. (8) A large, loose plug of asbestos; place the tube in the furnace, leaving about 2.5 cm. of it projecting; connect with the pump by a rubber stopper smeared with glycerol, taking care to make the connection perfectly tight.

OPERATION.

Exhaust the air from the tube by means of the pump. When a vacuum has been obtained allow the flow of mercury to continue; light the gas under that part of the tube containing the metallic copper, the anterior layer of cupric oxid (see 5th above), and the sodium bicarbonate. As soon as the vacuum is destroyed and the apparatus filled with carbonic acid, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer just below the surface of the mercury seal, so that the escaping bubbles will pass into the air and not into the tube, thus avoiding the useless saturation of the caustic-potash solution.

When the flow of carbonic acid has very nearly or completely ceased, pass the delivery tube down into the receiving arm, so that the bubbles will escape into the azotometer. Light the gas under the 30 cm. layer of oxid, heat gently for a few moments to drive out any moisture that may be present, and bring to red heat. Heat gradually the mixture of substance and oxid, lighting one jet at a time. Avoid a too rapid evolution of bubbles, which should be allowed to escape at the rate of about one per second or a little faster.

When the jets under the mixture have all been turned on, light the gas under the layer of oxid at the end of the tube. When the evolution of gas has ceased, turn out all the lights except those under the metallic copper and anterior layer of oxid, and allow to cool for a few moments. Exhaust with the pump and remove the azotometer before the flow of mercury is stopped. Break the connection of the tube with the pump, stop the flow of mercury, and extinguish the lights. Allow the azotometer to stand for at least an hour, or cool with a stream of water until a permanent volume and a temperature have been reached.

Adjust accurately the level of the KOH solution in bulb to that in the azotometer; note the volume of gas, temperature, and height of barometer; make calculations as usual, or read results from tables.

[Not applicable in presence of nitrates.]

REAGENTS.

(1 *Acid.*—Standard hydrochloric acid, the absolute strength of which has been determined: (*a*) By precipitating with silver nitrate and weighing the silver chlorid as follows:

Weigh the solution in a light glass-stoppered bottle, and turn it into a deep porcelain capsule, about 125 cc. capacity, provided with a well-formed lip and handle. Rinse the bottle with 25 cc. distilled water. Add solution of silver nitrate in about the proportion of 25 cc. to 0.5 gram potassium chlorid, and 2 cc. pure nitric acid, specific gravity, 1.2. Heat to boiling point, and keep at this temperature for some minutes without allowing violent ebullition, and with constant stirring, until the precipitate assumes the granular form. Allow to cool somewhat, and then pass the fluid through the asbestos. Wash the precipitate by decantation, with 200 cc. of very hot water, to which has been added 8 cc. nitric acid and 2 cc. dilute solution of silver nitrate containing 1 gram of the salt in 100 cc. of water. The washing by decantation is performed by adding the hot mixture in small quantities at a time, and beating up the precipitate well with a thin glass rod after each addition. The pump is kept in action all the time, but to keep out dust during the washing the cover is only removed from the crucible when the fluid is to be added.

Put the capsule and precipitate aside, return the washings once through the asbestos so as to obtain them quite clear, remove them from the filter, and set aside to recover excess of silver. Rinse the receiver and complete the washing of the precipitate with about 200 cc. of cold water. Half of this is used to wash by decantation, and the remainder to transfer the precipitate to the crucible with the aid of a trimmed feather. Finish washing in the crucible, the lumps of silver chlorid being broken down with the glass rod. Remove the second filtrate from the receiver and pass about 20 cc. of 98 per cent alcohol through the precipitate. Dry at 140° to 150°. Exposure for half an hour is found more than sufficient, at this temperature, to dry the precipitate thoroughly.

For ordinary work, half normal acid, *i. e.*, acid containing 18.2285 grams of hydrochloric acid to the liter, is recommended; for work in determining very small amounts of nitrogen, one-tenth normal acid is recommended. In titrating hydrochloric acid and other mineral acids against ammonia solutions, use cochineal as indicator.

(2) *Standard ammonia,* the strength of which, relative to the acid, has been accurately determined. One-tenth normal ammonia solution, *i. e.*, containing 1.7051 grams of ammonia to the liter, is recommended for accurate work.

(3) *Sulphuric acid,* specific gravity 1.84, free from nitrates and also from ammonium sulphate, which is sometimes added in the process of manufacture to destroy oxids of nitrogen.

(4) *Metallic mercury* or mercuric oxid, HgO, prepared in the wet way. That prepared from mercuric nitrate can not be safely used.

(5) *Potassium permanganate* finely pulverized.

(6) *Granulated zinc,* pumice stone, or 0.5 gram of zinc dust are to be added to the contents of the flasks in distillation, when found necessary, in order to prevent bumping.

(7) *Potassium sulphid.*—A solution of 40 grams of commercial potassium sulphid in 1 liter of water.

(8) *Soda.*—A saturated solution of sodium hydrate free from nitrates.

(9) *Indicator.*—Solution of cochineal prepared as follows: Tincture of cochineal is prepared by digesting and frequently agitating 3 grams of pulverized cochineal in a

mixture of 50 cc. of strong alcohol with 200 cc. of distilled water, at ordinary temperatures, for a day or two. The solution is decanted or filtered through Swedish paper.

APPARATUS.

(1) *Kjeldahl digestion flasks* of hard, moderately thick, well-annealed glass. These flasks are about 22 cm. long, with a round, pear-shaped bottom, having a maximum diameter of 6 cm., and tapering out gradually in a long neck, which is 2 cm. in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cc.

(2) *Distillation flasks* of ordinary shape, of 550 cc. capacity, or preferably flasks of the same capacity, of pear-shaped bottom, of well-annealed glass, for both digestion and distillation, fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation. The bulbs are about 3 cm. in diameter, the tubes being of the same diameter as the condenser and cut off obliquely at the lower end. This is adjusted to the tube of the condenser by a rubber tube.

MANIPULATION.

(1) *The digestion.*—0.7 to 2.8 grams of the substance to be analyzed, according to its proportion of nitrogen, is brought into a digestion flask with approximately 0.7 gram of mercuric oxid, or its equivalent in metallic mercury, and 20 cc. of sulphuric acid. The flask is placed in an inclined position, and heated below the boiling point of the acid for from five to fifteen minutes, or until frothing has ceased. If the mixture froths badly, a small piece of paraffin may be added to prevent it. The heat is then raised until the acid boils briskly. No further attention is required till the contents of the flask have become a clear liquid, which is colorless, or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and while still hot, potassium permanganate is dropped in carefully and in small quantities at a time till, after shaking, the liquid remains of a green or purple color.

(2) *The distillation.*—After cooling, the contents of the flask are transferred to the distilling flask with about 200 cc. of water, with a few pieces of granulated zinc, pumice stone, or 0.5 gram of zinc dust when found necessary to keep the contents of the flask from bumping, and 25 cc. of potassium sulphid solution are added, shaking the flask to mix its contents. Next add 50 cc. of the soda solution, or sufficient to make the reaction strongly alkaline, pouring it down the side of the flask so that it does not mix at once with the acid solution. Connect the flask with the condenser, mix the contents by shaking, and distill until all ammonia has passed over into the standard acid. The first 150 cc. of the distillate will generally contain all of the ammonia. This operation usually requires from forty minutes to one hour and a half. The distillate is then titrated with standard ammonia.

The use of mercuric oxid in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in case of substances most difficult to oxidize, and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. The potassium sulphid removes all the mercury from the solution, and so prevents the formation of mercurammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen, and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present, which might otherwise escape notice.

ALTERNATE METHODS.

GUNNING METHOD.

MANIPULATION.

In a digestion flask holding from 250 to 500 cc. place from 0.7 to 2.8 grams of the substance to be analyzed, according to its proportion of nitrogen. Then add 10 grams of powdered potassium sulphate and from 15 to 25 cc. (ordinarily about 20 cc.) of concentrated sulphuric acid. Conduct the digestion as in the Kjeldahl process, starting with a temperature below boiling point and increasing the heat gradually until frothing ceases. Digest until colorless or nearly so. (Do not add either potassium permanganate or potassium sulphid.) Dilute, neutralize, and distill as in the Kjeldahl method. In neutralizing, it is convenient to add a few drops of phenolphthalein indicator, by which one can tell when the acid is completely neutralized, remembering that the pink color, which indicates an alkaline reaction, is destroyed by a considerable excess of strong fixed alkali. The distillation and titration are conducted as in the Kjeldahl method.

KJELDAHL METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.[1]

[Applicable to all fertilizers containing nitrates.]

Besides the reagents and apparatus given under the Kjeldahl method there will be needed—

(1) *Zinc dust.* This should be an impalpable power; granulated zinc or zinc filings will not answer.

(2) *Zinc sulphid.*

(3) *Commercial salicylic acid.*

THE DETERMINATION.

Bring from 0.7 to 1.4 grams of the substance to be analyzed into a Kjeldahl digesting flask, add to this 30 cc. of sulphuric acid containing 2 grams of salicylic acid, and shake thoroughly; then add gradually 2 grams of zinc dust, shaking the contents of the flask at the same time, or add to the substance 30 cc. of sulphuric acid containing 1 gram of salicylic acid; then add direct 2 grams of zinc sulphid or 5 grams of crystallized sodium thiosulphate. Finally place the flask on the stand for holding the digestion flasks, where it is heated over a low flame until all danger from frothing has passed. The heat is then raised until the acid boils briskly and the boiling continued until white fumes no longer pour out of the flask. This requires about 5 or 10 minutes. Add now approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury, and continue the boiling until the liquid in the flask is colorless or nearly so. (In case the contents of the flask are likely to become solid before this point is reached add 10 cc. more of sulphuric acid.) Complete the oxidation with a little potassium permanganate in the usual way and proceed with the distillation as described in the Kjeldahl method. The reagents should be tested by blank experiments.

RUFFLE METHOD.

REAGENTS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) A mixture of equal parts by weight of fine slaked lime and finely powdered sodium thiosulphate dried at 100°.

(3) A mixture of equal parts by weight of finely powdered granulated sugar and flowers of sulphur.

(4) Granulated soda-lime, as described under the soda-lime method.

[1] Described by Prof. M. A. Scovell.

APPARATUS.

(5) Combustion tubes of hard Bohemian glass, 30 cm. long and 1.3 cm. in diameter.

(6) Bulbed U-tubes or Will's bulbs, as described under the soda-lime method.

MANIPULATION.

(1) Clean the U-tube and introduce 10 cc. of standard acid.

(2) Fit the cork and glass connecting tube. Fill the tube as follows: (1) A loosely fitting plug of asbestos, previously ignited, and then 2.5 to 3.5 cm. of the thiosulphate mixture. (2) The weighed portion of the substance to be analyzed is intimately mixed with from 5 to 10 grams of the sugar and sulphur mixture. (3) Pour on a piece of glazed paper or in a porcelain mortar a sufficient quantity of thiosulphate mixture to fill about 25 cm. of tube; then add the substance to be analyzed, as previously prepared, mix carefully, and pour into the tube; shake down the contents of the tube; rinse off the paper or mortar with a small quantity of the thiosulphate mixture and pour into the tube; then fill up with soda-lime to within 5 cm. of the end of the tube. (4) Place another plug of ignited asbestos at the end of the tube and close with a cork. (5) Hold the tube in a horizontal position and tap on the table until there is a gas channel all along the top of the tube. Make connection with the U-tube containing the acid; aspirate and see that the apparatus is tight.

The combustion.—Place the prepared combustion tube in the furnace, letting the open end project a little, so as not to burn the cork. Commence by heating the soda-lime portion until it is brought to a full red heat. Then turn on slowly jet after jet toward the outer end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and the evolution of the gas has ceased and the liquid in the U-tube begins to recede toward the furnace attach the aspirator to the other limb of the U-tube, break off the end of the tube, and draw a current of air through for a few minutes. Detach the U-tube and wash the contents into a beaker or porcelain dish; add a few drops of the cochineal solution, and titrate.

SODA-LIME METHOD.

[Not applicable in presence of nitrates.]

REAGENTS AND APPARATUS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) Granulated soda-lime, fine enough to pass a 2.5 mm. sieve and thoroughly dry.

(3) Fine soda-lime, fine enough to pass a 1.25 mm. sieve, also thoroughly dry.

Excellent soda-lime may be easily and cheaply prepared by slaking 2½ parts of quicklime with a strong solution of 1 part of commercial caustic soda (such soda as is used in the Kjeldahl process), care being taken that there is enough water in the solution to slake the lime. The mixture is then dried and heated in an iron pot to incipient fusion, and when cold ground and sifted as above.

Instead of soda-lime Johnson's mixture of carbonate of soda and lime or slaked lime may be used.

Slaked lime may be granulated by mixing it with a little water to form a thick mass, which is dried in the water oven until hard and brittle. It is then ground and sifted as above. Slaked lime is much easier to work with than soda-lime and gives excellent results, though it is probable that more of it should be used in proportion to the substance to be analyzed than is the case with soda-lime.

(4) Asbestos which has been ignited and kept in a glass-stoppered bottle.

(5) Combustion tubes about 40 cm. long and of 12 mm. internal diameter, drawn out to a point and closed at one end.

(6) Large-bulbed U-tubes with glass stopcock, or Will's tubes with four bulbs,

MANIPULATION.

The substance to be analyzed should be powdered fine enough to pass through a sieve of 1 mm. mesh; 0.7 to 1.4 grams, according to the amount of nitrogen present, is taken for the determination. Into the closed end of the combustion tube put a small loose plug of asbestos, and upon it about 4 cm. of fine soda-lime. In a porcelain dish or mortar mix the substance to be analyzed, thoroughly but quickly, with enough fine soda-lime to fill about 16 cm. of the tube, or about 40 times as much soda-lime as substance, and put the mixture into the combustion tube as quickly as possible by means of a wide-necked funnel, rinsing out the dish and funnel with a little more fine soda-lime, which is to be put in on top of the mixture. Fill the rest of the tube to about 5 cm. of the end with granulated soda-lime, making it as compact as possible by tapping the tube gently while held in a nearly upright position during the filling. The layer of granulated soda-lime should be not less than 12 cm. long. Lastly, put in a plug of asbestos about 2 cm. long, pressed rather tightly, and wipe out the end of the tube to free it from adhering soda-lime.

Connect the tube by means of a well-fitting rubber stopper or cork with the U-tube or Will's bulbs, containing 10 cc. of standard acid, and adjust it in the combustion furnace so that the end projects about 4 cm. from the furnace, supporting the U-tube or Will's bulb suitably. Heat the portion of the tube containing the granulated soda-lime to a moderate redness, and when this is attained extend the heat gradually through the portion containing the substance, so as to keep up a moderate and regular flow of gases through the bulbs, maintaining the heat of the first part until the whole tube is heated uniformly to the same degree. Keep up the heat until gases have ceased bubbling through the acid in the bulbs, and the mixture of substance and soda-lime has become white, or nearly so, which shows that the combustion is finished. The combustion should occupy about three-quarters of an hour or not more than one hour. Remove the heat, and when the tube has cooled below redness break off the closed tip and aspirate air slowly through the apparatus for two or three minutes to bring all the ammonia into the acid. Disconnect, wash the acid into a beaker or flask, and titrate with the standard alkali.

During the combustion the end of the tube projecting from the furnace must be kept heated sufficiently to prevent the condensation of moisture, yet not enough to char the stopper. The heat may be regulated by a shield of tin slipped over the projecting end of the combustion tube.

It is found very advantageous to attach a Bunsen valve to the exit tube, allowing the evolved gases to pass out freely, but preventing a violent "sucking back" in case of a sudden condensation of steam in the bulbs.

METHODS OF SUGAR ANALYSIS.[1]

DETERMINATION OF WATER.

In sugar or massecuite.—Dry from 2 to 5 grams in a flat dish (nickel or platinum) at the temperature of boiling water for three hours; cool in a desiccator and weigh; return to the oven and dry for an hour. If on weighing there is only a slight change of weight, the process may be considered finished; otherwise the drying must be continued until the loss of water in one hour is not greater than 0.20 per cent.

In honey or molasses.—Proceed as above, using only 1 to 2 grams of substance.

[1] By the term "sugar analysis" it is understood that the analyses of all the common sacharine substances, viz, juices, sugar, molasses, sirup, starch sugar, and honey, are embraced. In the following directions an attempt will be made to indicate the general principles which should guide the practice of sugar analysis, together with sufficient detail to enable these analyses to be carried out with accuracy and uniformity.

The principal components to be determined in the substances mentioned above are:

1. Water.
2. Density and total solids.
3. Ash.
4. Nitrogenous bodies.
5. Reducing sugar.
6. Sucrose.
7. Lactose.
8. Starch sugar.
9. Raffinose.

DENSITY AND TOTAL SOLIDS.

The density of juices, sirups, etc., is most conveniently determined by means of Baumé's or Brix's hydrometer or areometer, preferably with the latter, as the graduations of the scale give close approximations to the percentages of total solids. The Brix spindle should be graduated to tenths. It is therefore desirable, for accuracy, that the range of degrees recorded by each individual spindle be as limited as possible, this end being best secured by the employment of sets, consisting of not less than three spindles. The solutions should be as nearly as possible of the same temperature as the air at the time of reading, and if the variation from the standard temperature of the graduation of the spindle amounts to more than 1°, compensation therefor must be made by reference to tables of corrections for temperature.

Care should be observed in taking the density of a juice to allow it to stand in the cylinder until all air bubbles have escaped, before reading the spindles. When it is desired to accurately determine the specific gravity of the sample, a specific-gravity bottle or picnometer is employed.

The per cent of total solids can be approximately ascertained directly from the reading of the Brix hydrometer, or, knowing the reading of the Baumé spindle or the specific gravity of the sample, by reference to the table on page 214, bulletin 31.

DETERMINATION OF ASH.

(*a*) Heat 5 or 10 grams of the material (sugar, molasses, honey)[1] in a platinum dish[1] of 50 to 100 cc. capacity at 100° until the water is expelled, and then slowly over a flame until intumescence ceases. The dish is then placed in a muffle and heated at low redness until a white ash is obtained.

For soluble ash digest the ash with water, filter through a Gooch crucible, wash with hot water, dry the residue at 100°, and weigh. The difference of weights equals soluble ash.

(*b*) Use 50 milligrams of zinc oxid to 25 grams of molasses or 50 grams of sugar. Incorporate thoroughly by adding dilute alcohol and mixing; dry and ignite as above. Deduct the weight of zinc used from the weight of ash.

(*c*) Carbonize the mass at a low heat, dissolve the soluble salts with hot water, burn the residual mass as above, add the solution of soluble salts, and evaporate to dryness at 100°; ignite gently, cool in a desiccator, and weigh.

(*d*) Saturate the sample with H_2SO_4, dry, ignite gently, then burn in a muffle at low redness.

(*e*) Dissolve 10 grams of sugar in hot water and filter to remove sand, etc., evaporate the filtrate and washings to dryness; carefully carbonize and extract with hot water until there is no longer any reaction for chlorin. Dry and ignite the residue; weigh as insoluble ash. Add the soluble portion, treat with HCl in slight excess, and evaporate to dryness. Heat until all free HCl is driven off; take up with water and a little HCl, filter, and wash. The residue is silica. Add NH_4OH to the filtrate, boil and filter, wash the residue, which is iron oxid and alumina. To the filtrate add $(NH_4)_2C_2O_4$; evaporate to dryness; ignite and moisten with $(NH_4)_2CO_3$ and reignite; dissolve in water, filter, and wash. The residue on the filter represents calcium and magnesium carbonate. Evaporate the filtrate to a small bulk, add $(NH_4)_2CO_3$, and evaporate to dryness; drive off the excess of ammonia cautiously and weigh. This gives the alkalis as carbonates, and this weight, added to the insoluble ash, formerly determined, gives the total carbonate ash.

---

[1] If the substance contains tin or any other metal capable of uniting with platinum, a dish made of some other material must be used.

ALTERNATE METHOD FOR ASH.

To avoid the correction of one-tenth, as proposed by Scheibler, and one-fifth as proposed by Girard and Violette, when sugars are burned with H_2SO_4, Boyer suggests incineration with benzoic acid, as giving the real quantity of mineral matter without correction.

The benzoic acid is dissolved in alcohol of 90 per cent, 25 grams of the acid to 100 cc. of alcohol; 5 grams of the sugar are weighed in a capsule and moistened with 1 cc. of water. The capsule is heated slowly in order to caramelize the sugar without carbonizing it; 2 cc. of the benzoic-acid solution are next added, and the capsule warmed until all the alcohol is evaporated; the temperature is then raised until the sugar is converted into carbon. The decomposing benzoic acid produces abundant vapors, which render the mass extremely porous, especially if a circular motion is imparted to the capsule. The slow heating is continued until all the benzoic acid is volatilized.

The carbon obtained is voluminous and of a brilliant black color. The incineration is accomplished in a muffle at a low red heat. The capsule should be weighed quickly when taken from the desiccator, in order to avoid the absorption of water by the alkaline carbonates. Ammonium benzoate may be employed instead of benzoic acid, and the analyst should previously assure himself that neither the acid nor the ammonia salt leaves a residue on incineration. In addition to giving the mineral matters directly, this method permits the determination of their composition also, a matter of no small importance.

DETERMINATION OF NITROGEN.

Any of the methods adopted by the association for the estimation of nitrogen may be used.

REDUCING SUGARS.

Reagents (Soxhlet's modification of Fehling's solution).

(1). 34. 639 grams of $CuSO_4$, $5H_2O$, dissolved and diluted to 500 cc. with water; (2). 173 grams of Rochelle salt, 50 grams of NaOH, dissolved and diluted to 500 cc. with water. Mix equal volumes of these solutions immediately before use. This mixture is the "mixed copper reagent" to be used for all the methods given below except Allihn's method for dextrose, for which a special reagent must be used.

(*a*) *Approximate volumetric method for rapid work.*—Place 10 cc. of the mixed copper reagent in a large test tube and add 10 cc. of distilled water. Heat to boiling, and gradually add small portions of the solution of the material to be tested, boiling to complete the reaction after each addition, until the copper has been completely precipitated. Two minutes boiling is required for complete precipitation when the full amount of sugar solution has been added in one portion. When the end reaction is nearly reached and the amount of sugar solution to be added can no longer be judged by the color of the solution, a small portion of the liquid is removed by means of Knorr's modification of Wiley's filtering tube (any other rapid means of filtration may be used that removes but a small portion of the hot liquid), is transferred to a small porcelain crucible or test plate, acidified with dilute acetic acid, and tested for copper with a dilute solution of potassium ferrocyanid. The sugar solution should be of such a strength as will give a burette reading of 15–20 cc., and the number of successive additions should be as small as possible.

Since the factor for calculation varies with the minute details of manipulation, every operator must determine a factor for himself, using a known solution of a pure sample of the sugar that he desires to determine and keeping the conditions the same as those used for the determinations. For the standardization of a solution for the

determination of invert sugar in sugar-house products, dissolve 2.5 grams of pure sucrose in 100 cc. of water, add 10 cc. of hydrochloric acid (specific gravity 1.188), and invert according to the method given below for double polarization. Neutralize the acid with sodium carbonate and dilute to 1 liter. The 2.5 grams of sucrose become 2.6316 grams of invert sugar. The weight of invert sugar equivalent to 10 cc. of the copper reagent is calculated as follows:

$$\frac{2.6316 \times \text{by number of cc. of the standard sugar solution used}}{1000} = X,$$

the weight of invert sugar required to completely precipitate the copper in 10 cc. of the reagent under the conditions used for the test titration. For the calculation of the result of the titration of an unknown solution:

Let X=the factor obtained as above;

V=the number of cc. of unknown sugar solution required to precipitate the copper from 10 cc. of copper solution;

W=the weight of the material under examination in 1 cc. of the solution.

Then $\frac{100\ X}{V\ W}$=per cent of invert sugar in the sample.

The calculation can be much simplified by so standardizing the copper reagent that 50 milligrams of invert sugar will be required to reduce the copper from 10 cc. of the copper reagent. The various tables given in works on sugar analysis then become applicable. These tables are arranged for a "glucose normal solution" containing 5 grams of the material to be examined in 100 cc. When the weight per 100 cc. is more or less than 5 grams the number found in the table is increased or diminished accordingly.

(*b*) *Soxhlet's exact volumetric method.*—A preliminary titration is made to determine the approximate percentage of reducing sugar in the material under examination. A solution is then prepared which contains approximately 1 per cent of reducing sugar. Place in a beaker 100 cc. of the mixed copper reagent and approximately the amount of the sugar solution for its complete reduction. Boil for two minutes. Filter through a plaited filter and test a portion of the filtrate for copper by use of acetic acid and potassium ferrocyanid. Repeat the test, varying the volume of sugar solution, until two successive amounts of sugar solution are found, which differ by 0.1 cc., one giving complete reduction and the other leaving a small amount of copper in solution. The mean of these two readings is taken as the volume of the solution required for the complete precipitation of 100 cc. of the copper reagent.

Under these conditions 100 cc. of the mixed copper reagent require 0.475 gram of anhydrous dextrose, or 0.494 gram of invert sugar for complete reduction. The percentage is calculated by the following formula:

W = the weight of the sample in 1 cc. of the sugar solution;

V = the volume of the sugar solution required for the complete reduction of 100 cc. of the copper reagent.

Then $\frac{100 \times 0.475}{V\ W}$ = per cent of dextrose,

or $\frac{100 \times 0.494}{V\ W}$ = per cent of invert sugar.

(*c*) *Gravimetric method for materials containing 1 per cent or less of invert sugar.*—The solution of the material to be examined is so prepared as to contain 20 grams in 100 cc., and it must be freed from suspended impurities by filtration through paper or by use of lead acetate, removing the excess of lead by means of sodium carbonate. In a beaker of 250 cc. capacity place 50 cc. of the mixed copper reagent and 50 cc. of the sugar solution. Heat this mixture at such a rate that approximately four minutes are required to bring it to the boiling point and boil for exactly two minutes. Add 100 cc. of cold, recently boiled, distilled water. Filter immediately through a

weighed filtering tube of hard glass under pressure. The asbestos film in the filtering tube is supported by a perforated disk or cone of platinum and should be washed free from loose fibers before weighing and moistened previous to the filtration. The tube is provided with a detachable funnel during the filtration so that none of the precipitate accumulates near the top, where it could be removed by the cork used during the reduction of the cuprous oxid. The precipitate is all transferred to the filter and thoroughly washed with hot water, following the water by alcohol and ether successively. After being dried the tube is connected with an apparatus for supplying a continuous current of dry hydrogen, gently heated until the cuprous oxid is completely reduced to the metallic state, cooled in the current of hydrogen, and weighed. The increase in weight is the weight of copper reduced by 10 grams of the sample. The corresponding percentage of invert sugar is found by use of the following table:

Herzfeld's table for the determination of invert sugar in materials containing 1 per cent or less of invert sugar.[1]

Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.
Milligrams.	*Per cent.*	*Milligrams.*	*Per cent.*	*Milligrams.*	*Per cent.*
50	0.05	120	0.40	190	0.79
55	0.07	125	0.43	195	0.82
60	0 09	130	0.45	200	0.85
65	0.11	135	0.48	205	0.88
70	0.14	140	0.51	210	0.90
75	0.16	145	0.53	215	0.93
80	0.19	150	0.56	220	0.96
85	0.21	155	0.59	225	0.99
90	0.24	160	0.62	230	1.02
95	0.27	165	0.65	235	1.05
100	0.30	170	0.68	240	1.07
105	0.32	175	0.71	245	1.10
110	0.35	180	0.74		
115	0.38	185	0.76		

[1] Zeitschrift des Vereins für die Rübenzucker-Industrie des Deutschen Reichs, 1885, 35, 967.

Alternate method for obtaining the weight of the copper.—The filtration after reduction is made in a Gooch crucible, and the beaker and precipitate thoroughly washed with hot water without any effort to transfer the precipitate to the filter. Wash the asbestos film and the adhering cuprous oxid into the beaker by means of a wash bottle containing hot dilute nitric acid. After the copper is all in solution refilter through a Gooch crucible with a thin film of asbestos and wash thoroughly with hot water. Add 10 cc. of dilute sulphuric acid (containing 200 cc. of sulphuric acid—specific gravity 1.84—per liter) and evaporate the filtrate on the steam bath until the copper salt has largely crystallized out. Heat carefully on a hot plate or over a piece of asbestos board until the evolution of white fumes shows that the excess of nitric acid is removed. Add 8 to 10 drops of nitric acid (specific gravity 1.42) and rinse into a platinum dish of 100 to 125 cc. capacity. Precipitate the copper by electrolysis. Wash with water thoroughly before breaking the current, remove the dish from the circuit, wash with alcohol and ether successively, dry at a temperature that can be borne by the hand, and weigh. The evaporation of the liquid for the removal of the excess of nitric acid may be avoided by the following process: Transfer the asbestos film from the crucible to the beaker by means of a glass rod and rinse the crucible with about 30 cc. of a boiling mixture of dilute nitric and sulphuric acids, containing 65 cc. of sulphuric acid (specific gravity 1.84) and 50 cc. of nitric acid

(specific gravity 1.42) per liter; or the asbestos film with the adhering cuprous oxid may be treated by the method of Formanek given below, by transferring to the beaker containing the rest of the suboxid and washing the crucible with 2–4 cc. of nitric acid (strong) to remove all adhering precipitate, receiving the solutions in the beaker. The crucible is then washed with 5–10 cc. water which is also received in the beaker. After all the cuprous oxid in the beaker is dissolved the crucible is worked with an additional quantity of water. Refilter through a Gooch crucible and wash thoroughly with hot water, limiting the volume of the filtrate to 75 to 80 cc. Transfer to a platinum dish and electrolyze as described above. Wash before interrupting the current, using water acidulated with sulphuric acid at first.

(*d*) *Gravimetric method for materials containing more than 1 per cent of invert sugar.*—Prepare a solution of the material to be examined in such a manner that it contains 20 grams in 100 cc. after clarification and the removal of the excess of lead. Prepare a series of solutions in large test tubes by adding 1, 2, 3, 4, 5, etc., cc. of this solution to each successively. Add 5 cc. of the mixed copper reagent to each, heat to boiling, boil two minutes, and filter. Note the volume of sugar solution which gives the filtrate lightest in tint, but still distinctly blue. Place twenty times this volume of the sugar solution in a 100 cc. flask, dilute to the mark, and mix well. Use 50 cc. of the solution for the determination, which is conducted as described under *c*, until the weight of copper is obtained. For the calculation of the result use the following formulas and table of factors of Meissl and Hiller:

Let Cu = the weight of copper obtained;
P = the polarization of the sample;
W = the weight of the sample in the 50 cc. of the solution used for determination;
F = the factor obtained from the table for conversion of copper to invert sugar;

$\frac{Cu}{2}$ = approximate absolute weight of invert sugar = Z;

$Z \times \frac{100}{W}$ = approximate per cent of invert sugar = y;

$\frac{100\ P}{P+y}$ = R, relative number for sucrose;

100 − R = I, relative number for invert sugar;

$\frac{Cu\ F}{W}$ = per cent of invert sugar.

Z facilitates reading the vertical columns; and the ratio of R to I, the horizontal columns of the table, for the purpose of finding the factor (F) for calculation of copper to invert sugar.

Example: The polarization of a sugar is 86.4, and 3.256 grams of it (W) are equivalent to 0.290 grams of copper. Then:

$$\frac{Cu}{2} = \frac{.290}{2} = 0.145 = Z$$

$$Z \times \frac{100}{W} = 0.145 \times \frac{100}{3.256} = 4.45 = Y$$

$$\frac{100\ P}{P+y} = \frac{8640}{86.4+4.45} = 95.1 = R$$

$$100 - R = 100 - 95.1 = I = 4.9$$

$$R : I = 95.1 : 4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95:5 is nearest to the ratio of R to I, 95.1:4.9. Where these columns meet we find the factor 51.2, which enters into the final calculation:

$$\frac{Cu\ F}{W} = \frac{0.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert sugar.}$$

Meissl and Hiller's factors for the determination of more than 1 per cent of invert sugar.[1]

Ratio of sucrose to invert sugar = R : I.	Approximate absolute weight of invert sugar = Z.						
	200 milligrams.	175 milligrams.	150 milligrams.	125 milligrams.	100 milligrams.	75 milligrams.	50 milligrams.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
0 : 100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10 : 90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20 : 80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30 : 70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40 : 60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50 : 50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60 : 40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70 : 30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80 : 20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90 : 10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91 : 9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92 : 8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93 : 7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94 : 6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95 : 5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

[1] Zeitschrift des Vereins für der Rübenzucker-Industrie des Deutschen Reichs, 1889, **39**, 735.

(*e*) *Allihn's gravimetric method for the determination of dextrose.*

Reagents:

I. 34.639 grams of $CuSO_4, 5H_2O$, dissolved in water and diluted to 500 cc.

II. 173 grams of Rochelle salts, 125 grams of KOH, } dissolved in water and diluted to 500 cc.

Place 30 cc. of the copper solution (I), 30 cc. of the alkaline tartrate solution (II), and 60 cc. of water in a beaker and heat to boiling. Add 25 cc. of the solution of the material to be examined, which must be so prepared as not to contain more than 1 per cent of dextrose, and boil for two minutes. Filter immediately without diluting and obtain the weight of copper by one of the methods given under (*c*). The corresponding weight of dextrose is found by the following table:

Allihn's table for the determination of dextrose.[1]

Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
10	6.1	22	12.0	34	18.0	46	23.9	58	29.8
11	6.6	23	12.5	35	18.5	47	24.4	59	30.3
12	7.1	24	13.0	36	18.9	48	24.9	60	30.8
13	7.6	25	13.5	37	19.4	49	25.4	61	31.3
14	8.1	26	14.0	38	19.9	50	25.9	62	31.8
15	8.6	27	14.5	39	20.4	51	26.4	63	32.3
16	9.0	28	15.0	40	20.9	52	26.9	64	32.8
17	9.5	29	15.5	41	21.4	53	27.4	65	33.3
18	10.0	30	16.0	42	21.9	54	27.9	66	33.8
19	10.5	31	16.5	43	22.4	55	28.4	67	34.3
20	11.0	32	17.0	44	22.9	56	28.8	68	34.8
21	11.5	33	17.5	45	23.4	57	29.3	69	35.3

[1] J. prakt. Chem., 1880, **22**, 46.

Allihn's table for the determination of dextrose[1]—Continued.

Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
70	35.8	122	62.1	174	89.0	226	116.4	278	144.4
71	36.3	123	62.6	175	89.5	227	116.9	279	145.0
72	36.8	124	63.1	176	90.0	228	117.4	280	145.5
73	37.3	125	63.7	177	90.5	229	118.0	281	146.1
74	37.8	126	64.2	178	91.1	230	118.5	282	146.6
75	38.3	127	64.7	179	91.6	231	119.0	283	147.2
76	38.8	128	65.2	180	92.1	232	119.6	284	147.7
77	39.3	129	65.7	181	92.6	233	120.1	285	148.3
78	39.8	130	66.2	182	93.1	234	120.7	286	148.8
79	40.3	131	66.7	183	93.7	235	121.2	287	149.4
80	40.8	132	67.2	184	94.2	236	121.7	288	149.9
81	41.3	133	67.7	185	94.7	237	122.3	289	150.5
82	41.8	134	68.2	186	95.2	238	122.8	290	151.0
83	42.3	135	68.8	187	95.7	239	123.4	291	151.6
84	42.8	136	69.3	188	96.3	240	123.9	292	152.1
85	43.4	137	69.8	189	96.8	241	124.4	293	152.7
86	43.9	138	70.3	190	97.3	242	125.0	294	153.2
87	44.4	139	70.8	191	97.8	243	125.5	295	153.8
88	44.9	140	71.3	192	98.4	244	126.0	296	154.3
89	45.4	141	71.8	193	98.9	245	126.6	297	154.9
90	45.9	142	72.3	194	99.4	246	127.1	298	155.4
91	46.4	143	72.9	195	100.0	247	127.6	299	156.0
92	46.9	144	73.4	196	100.5	248	128.1	300	156.5
93	47.4	145	73.9	197	101.0	249	128.7	301	157.1
94	47.9	146	74.4	198	101.5	250	129.2	302	157.6
95	48.4	147	74.9	199	102.0	251	129.7	303	158.2
96	48.9	148	75.5	200	102.6	252	130.3	304	158.7
97	49.4	149	76.0	201	103.1	253	130.8	305	159.3
98	49.9	150	76.5	202	103.7	254	131.4	306	159.8
99	50.4	151	77.0	203	104.2	255	131.9	307	160.4
100	50.9	152	77.5	204	104.7	256	132.4	308	160.9
101	51.4	153	78.1	205	105.3	257	133.0	309	161.5
102	51.9	154	78.6	206	105.8	258	133.5	310	162.0
103	52.4	155	79.1	207	106.3	259	134.1	311	162.6
104	52.9	156	79.6	208	106.8	260	134.6	312	163.1
105	53.5	157	80.1	209	107.4	261	135.1	313	163.7
106	54.0	158	80.7	210	107.9	262	135.7	314	164.2
107	54.5	159	81.2	211	108.4	263	136.2	315	164.8
108	55.0	160	81.7	212	109.0	264	136.8	316	165.3
109	55.5	161	82.2	213	109.5	265	137.3	317	165.9
110	56.0	162	82.7	214	110.0	266	137.8	318	166.4
111	56.5	163	83.3	215	110.6	267	138.4	319	167.0
112	57.0	164	83.8	216	111.1	268	138.9	320	167.5
113	57.5	165	84.3	217	111.6	269	139.5	321	168.1
114	58.0	166	84.8	218	112.1	270	140.0	322	168.6
115	58.6	167	85.3	219	112.7	271	140.6	323	169.2
116	59.1	168	85.9	220	113.2	272	141.1	324	169.7
117	59.6	169	86.4	221	113.7	273	141.7	325	170.3
118	60.1	170	86.9	222	114.3	274	142.2	326	170.9
119	60.6	171	87.4	223	114.8	275	142.8	327	171.4
120	61.1	172	87.9	224	115.3	276	143.3	328	172.0
121	61.6	173	88.5	225	115.9	277	143.9	329	172.5

[1] J. prakt. Chem., 1880, 22, 46.

Allihn's table for the determination of dextrose[1]—Continued.

Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
330	173.1	357	188.3	384	203.7	411	219.3	438	235.1
331	173.7	358	188.9	385	204.3	412	219.9	439	235.7
332	174.2	359	189.4	386	204.8	413	220.4	440	236.3
333	174.8	360	190.0	387	205.4	414	221.0	441	236.9
334	175.3	361	190.6	388	206.0	415	221.6	442	237.5
335	175.9	362	191.1	389	206.5	416	222.2	443	238.1
336	176.5	363	191.7	390	207.1	417	222.8	444	238.7
337	177.0	364	192.3	391	207.7	418	223.3	445	239.3
338	177.6	365	192.9	392	208.3	419	223.9	446	239.8
339	178.1	366	193.4	393	208.8	420	224.5	447	240.4
340	178.7	367	194.0	394	209.4	421	225.1	448	241.0
341	179.3	368	194.6	395	210.0	422	225.7	449	241.6
342	179.8	369	195.1	396	210.6	423	226.3	450	442.2
343	180.4	370	195.7	397	211.2	424	226.9	451	242.8
344	180.9	371	196.3	398	211.7	425	227.5	452	243.4
345	181.5	372	196.8	399	412.3	426	228.0	453	244.0
346	182.1	373	197.4	400	212.9	427	228.6	454	244.6
347	182.6	374	198.0	401	213.5	428	229.2	455	245.2
348	183.2	375	198.6	402	214.1	429	229.8	456	245.7
349	183.7	376	199.1	403	214.6	430	230.4	457	246.3
350	184.3	377	199.7	404	215.2	431	231.0	458	246.9
351	184.9	378	200.3	405	215.8	432	231.6	459	247.5
352	185.4	379	200.8	406	216.4	433	232.2	460	248.1
353	186.0	380	201.4	407	217.0	434	232.8	461	248.7
354	186.6	381	202.0	408	217.5	435	233.4	462	249.3
355	187.2	382	202.5	409	218.1	436	233.9	463	249.9
356	187.7	383	203.1	410	218.7	437	234.5		

[1] J. prakt. Chem., 1880, 22, 46.

Modified method of Formanek for electrolytic estimation of reduced copper (for provisional use).—To the solution obtained by the treatment of the precipitated suboxid with nitric acid and hot water add a sufficient quantity of water to reduce the nitric acid content of the solution below 5 per cent, and electrolyze.

Instead of dissolving the suboxid with nitric acid on the filter, the asbestos filter and contents are transferred from the funnel to the beaker used in the precipitation, and the funnel is washed out with a 4 per cent nitric-acid solution. This acid solution is added to the contents of the beaker until a bulk of about 200 cc. is attained.

Two platinum cylinders are then immersed in the liquid and connections are made with a battery, the larger cylinder being the cathode.

OFFICIAL ESTIMATE OF SUCROSE.

(*a*) *In sugars, massecuites, etc.*—Take the normal weight in tared dish for the instrument employed; wash into a 100 cc. flask; add water until volume is 80 to 85 cc. When the crystals are all dissolved add sufficient lead acetate to throw down all precipitable matter. With molasses and massecuites add sufficient acetic acid to convert the sub into the neutral acetate. Make up to mark, using a little ether spray to dissolve bubbles; filter, throwing away the first 10 to 15 cc.; place in observation tube and polarize. If too dark to read filter through finely powdered dry bone black, rejecting the first 30 to 40 cc.

For adjusting the polariscope, graduating flasks, etc., the method of the United

States Bureau of Internal Revenue, adopted last year, was continued. (See Bull. 31, pp. 228-232.)

(*b*) *In juices, etc.*—Transfer, by means of a pipette, to the tared sugar dish the normal, or multiple normal, weight of the juice or sirup to be analyzed.

In the case of juices and thin sirups, the contents of the dish are at once washed into the 100 cc. flask.

The solution in the flask is made up to 80 or 90 cc., and the least amount of basic lead-acetate solution sufficient to decolorize the contents, is added. The solution is then made up to the 100 cc. mark and thoroughly shaken before filtering. In case much foaming and bubbling takes place, rendering gauging difficult, the addition of a few drops of ether is found advantageous. The solution is then poured upon a dry filter and the filtrate is collected in a dry beaker, the first portions to pass through being rejected. If it is found impossible to obtain a perfectly clear filtrate it may be necessary to decolorize with dry powdered bone black, either mixed with the liquid itself or placed upon the filter.

In case of dark-colored products, the addition of alumina cream immediately after the lead subacetate is quite advantageous, the quantity of the former employed being in excess of that of the latter. For many sugars the use of alumina cream alone will produce a satisfactory clarification. Where double polarization is employed, acetic acid should be added to the filtrate used for the direct reading, in sufficient proportion to break up the compound of lead and levulose which has been formed.

Preparation of lead subacetate solution.—Boil an aqueous solution of lead acetate with an excess of lead oxid (PbO) for half an hour, and make filtered solution of a concentration of not less than 1.25 specific gravity. Solid subacetate of lead may be substituted for the normal salt and oxide in the preparation of the solution.

Alumina cream.—Prepare a cold saturated solution of alum in water and divide into two unequal portions. Add a slight excess of ammonium hydrate to the larger portion, and then add by degrees the remaining alum solution until a faintly acid reaction is secured.

Optical method by inversion.—For raw sugar, molasses, etc.

(*a*) *Method of Clerget.*—Make up the solution as above, and place 50 cc. of the filtrate in a flask marked at 50 and 55 cc. Fill to the upper mark with pure fuming HCl and mix well; place in water and heat until the thermometer, with the bulb as near the center of the sugar solution in the flask as possible, marks 68°, consuming about ten minutes in the heating; remove, cool quickly to room temperature, and polarize, noting the temperature. If the sample contained originally any invert sugar the second polarization should be made at approximately the same temperature as the first. The percentage of sucrose is then calculated by the following formula:

S = percentage of sucrose.
a = first polarization.
b = second polarization (usually to the left).
$a \pm b$ = sum of the polarizations.
t = temperature of observation.

Then

$$S = \frac{a \pm b}{144 - \frac{t}{2}}$$

a and b are to be added when of different signs and the difference taken when of the same sign.

(*b*) *The official German method* may be used as follows: 26.048 grams are dissolved in a sugar flask and the solution made up to 100 cc.; 50 cc. of this solution are transferred by means of a pipette to a 50 or 55 cc. flask, cleared, and polarized, the reading being corrected for the extra 5 cc. The liquid adhering to the pipette is washed into the 100 cc. flask containing the remaining 50 cc. (13.024 grams), 5 cc. of concen-

trated hydrochloric acid (38 per cent, specific gravity 1.18 at 15°) added and the flask heated for fifteen minutes at 67° to 70° in a water bath. The temperature should not exceed this limit. The flask is then cooled rapidly and the solution made up to 100 cc. If the liquid is colored it is shaken with ½ to 1 gram of bone black and filtered through a double filter. It is then polarized in a 200 mm. tube which is provided with a thermometer. As the rotatory power of invert sugar is much influenced by the temperature, this factor must be regarded. The reading is best taken between 18° and 22° and the temperature accurately determined. The reading must also be doubled on account of the dilution of the liquid.

To calculate per cent of sucrose (R) the two readings are added together, the sum (S) multiplied by 100 and divided by $142.4 - \frac{1}{2}t$ where t is the temperature at which the invert reading was taken. If the temperature is exactly 20° the result can be made more accurate by substituting 142.66 for 142.4; thus:

$$R = \frac{100\ S}{142.66 - \frac{2}{2}^{0}} = \frac{100\ S}{132.66} = 0.7538\ S.$$

If very much invert sugar is present, both the direct and indirect readings must be taken at the same temperature.

(*c*) *Method of Lindet.*—Place 50 cc. of the filtrate as above in a flask marked at 100.5 cc.; add 5 grams of powdered zinc (zinc dust), place in boiling water, and when at the boiling point add, little by little, 5 cc. of strongest HCl. After the acid has all been added, cool and make up to mark. Polarize in a 400-mm. tube, or multiply the reading in a 200-mm. tube by 2. Calculate the percentage of sucrose as before.

(*d*) *Gravimetric method.*—Determine first any reducing sugar in the sample; then invert sucrose, neutralize free acid, and redetermine the reducing sugar. Deduct the percentage of reducing sugar obtained at first, and the remainder will be reducing sugar derived from sucrose; multiply this number by .95 to obtain the percentage of sucrose in the sample.

ELECTIVE METHODS.

The elective methods remain the same as in Bulletin 28, pp. 217–228.

METHODS OF ANALYSIS OF FOODS AND FEEDING STUFFS.

PREPARATION OF SAMPLE.

The substance is to be ground and passed through a sieve with circular holes 1 millimeter in diameter.

DETERMINATION OF MOISTURE.

Dry from 2 to 3 grams of the substance for five hours, at the temperature of boiling water, in a current of dry hydrogen. If the substance be held in a glass vessel the latter should not be in contact with the boiling water.

DETERMINATION OF ASH.

Char 2 to 3 grams of the substance and burn to whiteness at the lowest possible red heat. If a white ash can not be obtained in this manner, exhaust the charred mass with water; collect the insoluble residue on a filter, burn, add this ash to the residue from the evaporation of the above aqueous extract, and heat the whole to a low redness till the ash is white.

DETERMINATION OF ETHER EXTRACT.

Extract 2 to 3 grams of the substance dried as for the determination of the moisture, with anhydrous and alcohol-free ether, sixteen hours. Dry the extract, by exposure to the full heat of boiling water, to constant weight.

ALTERNATE METHOD FOR ETHER EXTRACT.

In determining hygroscopic water, as above, continue the drying until the loss of weight in 30 minutes is reduced to 1 milligram or less; extract the dried substance for sixteen hours as directed, dry again, and give loss of weight as ether extract.

Anhydrous ether.—To prepare the anhydrous alcohol-free ether required for estimation of fat, take any of the commercial brands of ether, wash with two or three successive portions of distilled water, add sticks of solid caustic soda or potash until most of the water has been abstracted from the ether. Carefully cleaned metallic sodium, cut into small pieces, is now added until there is no further evolution of hydrogen gas. The ether thus dehydrated must be kept over metallic sodium, and should be only lightly stoppered in order to allow any accumulating hydrogen gas to escape; and it may be drawn off with a pipette as required.

CRUDE PROTEIN.

Determine nitrogen as directed for nitrogen in fertilizers and multiply the result by 6.25 for the crude protein.

DETERMINATION OF ALBUMINOID NITROGEN—STUTZER'S METHOD.

To 0.7 to 0.8 gram of the substance add 100 cc. of water in a beaker, heat to boiling, or in the case of substances rich in starch heat on the water bath ten minutes, add a quantity of cupric hydrate mixture containing 0.5 to 0.6 gram of the hydrate stir thoroughly, filter when cold, wash with cold water, and put the filter and its contents into the concentrated sulphuric acid for the determination of nitrogen. The filter papers used must be practically free of nitrogen. Add sufficient potassium-sulphid solution to completely precipitate all copper and mercury. If the substance examined consist of seed of any kind, or residues of seeds, such as oil cake or anything else rich in alkaline phosphates, add a few cc. of a concentrated solution of alum just before adding the cupric hydrate, and mix well by stirring. This serves to decompose the alkaline phosphates. If this is not done cupric phosphate and free alkali may be formed, and the protein-copper may be partially dissolved in the alkaline liquid.

Cupric hydrate.—Prepare cupric hydrate as follows: Dissolve 100 grams of pure cupric sulphate in 5 liters of water, and add 25 cc. of glycerol; add dilute solution of sodium hydrate until the liquid is alkaline; filter; rub the precipitate up with water containing 5 cc. of glycerol per liter, and then wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent of glycerol, thus preparing a uniform gelatinous mass that can be measured out with a pipette. Determine the quantity of cupric hydrate per cubic centimeter of this mixture.

DETERMINATION OF CRUDE FIBER.

(*1*) *In feeding stuffs poor in starch.*—Extract 2 grams of the substance with ordinary ether, at least nearly completely, or take the residue from the determination of the ether extract. To this residue, in a 500 cc. flask, add 200 cc. of boiling 1.25 per cent sulphuric acid; connect the flask with a return-flow condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once, and continue the boiling for thirty minutes. A blast of air conducted into the flask may serve to reduce the frothing of the liquid. Filter; wash thoroughly with boiling water till the washings are no longer acid; rinse the substance back into the same flask with 200 cc. of a boiling 1.25 per cent solution of sodium hydrate at least almost free from sodium carbonate; boil at once, and continue the boiling for thirty minutes in the same manner as directed above for the treatment with acid. Filter in a Gooch crucible, and wash with boiling water till the washings are neutral; dry at 110°; weigh; incinerate completely, and give the loss of weight for crude fiber.

The filter used for the first filtration may be linen, one of the forms of glass-wool or asbestos filters recommended in the last report, or any other form that secures clear and reasonably rapid filtration. The solutions of sulphuric acid and sodium hydrate are to be made up of the specified strength accurately by titration, and not merely from specific gravity.

(2) *In feeding stuffs rich in starch.*—Proceed as under (1) using 2.5 per cent acid and alkali in place of the solution of 1.25 per cent strength.

METHODS OF ANALYSIS OF DAIRY PRODUCTS.

BUTTER.

MICROSCOPIC EXAMINATION.

Place a small portion of the fresh samples, taken from the inside of the mass, on a slide, add a drop of pure sweet oil, cover with gentle pressure, and examine with a one-half to one-eighth inch objective for crystals of lard, etc. Examine the same specimen with polarized light and a selenite plate without the use of oil. Pure fresh butter will neither show crystals nor a particolored field with selenite. Other fats melted and cooled and mixed with butter will usually present crystals and variegated colors with the selenite plate.

For further microscopic study dissolve 4 or 5 cc. of the fat in 15 cc. of ether in a test tube. Close the tube with a loose plug of cotton wool and allow to stand 12 to 24 hours at room temperature (20° to 25°). When crystals form at the bottom of the tube they are removed with a pipette, glass rod, or tube, placed on a slide, covered, and examined. The crystals formed by later deposits may be examined in a similar way.

SAMPLING.

If large quantities of butter are to be sampled a butter trier or sampler may be used. The portions thus drawn, about 500 grams, are to be perfectly melted in a closed vessel at as low a heat as possible, and when melted the whole is to be shaken violently for some minutes till the mass is homogeneous.[1] A portion is then poured into the vessel from which it is to be weighed for analysis, and should nearly or quite fill it. This sample should be kept in a cold place till analyzed.

DETERMINATION OF WATER.

One and five-tenths to 2.5 grams are dried to constant weight at the temperature of boiling water in a dish with flat bottom, having a surface of at least 20 cm^2.

The use of clean dry sand or asbestos with the butter is admissible, and is necessary if a dish with round bottom is employed.

DETERMINATION OF FAT.

The dry butter from the water determination is dissolved in the dish with absolute ether, or with 76° benzine. The contents of the dish are then transferred to a weighed Gooch crucible with the aid of a wash bottle filled with the solvent, and are washed till free from fat. The crucible and contents are heated at the temperature of boiling water till the weight is constant. The weight of fat is calculated from the data obtained.

ALTERNATE METHOD FOR FAT.

Water may be determined by drying the butter on asbestos or sand, and the fat extracted by anhydrous alcohol-free ether. The extract, after evaporation of the ether, is heated to constant weight at the temperature of boiling water and weighed.

[1] The mass must be sufficiently solidified to prevent the separation of the water and fat after it is placed in the bottle.

DETERMINATION OF CASEIN AND ASH.

The crucible containing the residue from the fat determination, consisting of casein and ash, is covered and heated, gently at first, gradually raising the temperature to just below redness. The cover may then be removed and the heat continued till the contents of the crucible are white. The loss in weight of the crucible and contents represents casein, and the residue in the crucible mineral matter. In this mineral matter, dissolved in water slightly acidulated with nitric acid, chlorin may be determined gravimetrically with silver nitrate, or volumetrically, using potassium chromate as an indicator.

DETERMINATION OF SALT.

The amount of the butter or butter substitute to be taken is from 5 to 10 grams; weigh in a counterpoised beaker-glass. The butter is placed, in portions of about 1 gram at a time, in the beaker, these portions being taken from different parts of the sample. Hot water is now added (about 20 cc.) to the beaker containing the butter, and after it has melted the liquid is poured into the bulb of a separating funnel. The stopper is now inserted and the contents shaken for a few moments. After standing until the fat has all collected on top of the water, the stopcock is opened and the water is allowed to run into an Erlenmeyer flask, being careful to let none of the fat globules pass. Hot water is again added to the beaker, and the foregoing process is repeated from 10 to 15 times, using each time 10 to 20 cc. of water. The resulting washings contain all but a mere trace of the NaCl originally present in the butter. The sodium chlorid is now determined in the filtrate by a standard solution of $AgNO_3$, using a few drops of a saturated solution of potassium chromate as an indicator.

SPECIFIC GRAVITY.

Standardization of flasks.—Use a small specific-gravity flask of from 25 to 30 cc. capacity. The flask is to be thoroughly washed with hot water, alcohol, and ether, and then dried. After cooling in a desiccator, the weight of the flask and stopper is accurately determined.

The flask is filled with freshly boiled and still hot distilled water, and placed in a bath of pure distilled water. The water of the bath is kept in brisk ebullition for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. The stopper[1] is then inserted, the flask removed, wiped dry, and, after it is nearly cooled to room temperature, placed in the balance, and weighed when balance temperature is reached.

Weight of fat at the temperature of boiling water.—The flask is emptied of its water, rinsed with alcohol and ether, and dried again for a few minutes at the temperature of boiling water. It is then filled with the dry, hot, fresh-filtered fat, which should be be entirely free from air bubbles, and replaced in the water bath, kept for thirty minutes at the temperature of boiling water, removed, and treated as above. The weight of fat having been determined, the specific gravity is obtained by dividing it by the weight of water previously found.

Example.

	Grams.
Weight of flask No. 22, dry	10.0197
Weight of flask No. 22, plus water	37.3412
Weight of water	27.3215
Weight of flask No. 22, plus fat	34.6111
Weight of fat	24.5914

Specific gravity = 24.5914 ÷ 27.3215 = .90008.

[1] The stopper should be kept for a few minutes before use in hot distilled water.

The weight of the flask dry and empty, and the weight of water at boiling temperature contained therein, may be used constantly if great care be taken in handling and cleaning the apparatus.

Example.

	Grams.
Weight of flask No. 10, dry and empty	10.0028
Weight of flask after three weeks' use	10.0030

Alternate method of standardizing flasks.—Formula for calculating the volume V, in cubic centimeters, of a glass vessel from the weight P of water of temperature t contained therein, and the volume V′ at any other temperature t' (from Landolt and Börnstein's physical-chemical tables, p. 39, Table 17):

$$V = P\frac{p}{d}$$

$$V' = P\frac{p}{d}\left[1 + \gamma\,(t' - t)\right]$$

p = weight (in brass weights) of 1 cc. of H_2O in vacuo. (This is so nearly 1 that it will not affect the result in the fifth place of decimals, and may therefore be disregarded.) Hence the formula stands:

$$V' = P\frac{1}{d}\left[1 + \gamma\,(t' - t)\right]$$

d = density of water at temperature t.

γ = .000025, the cubical expansion coefficient of glass.

MELTING POINT.

The apparatus for determining the melting point consists of (1) an accurate thermometer for reading easily tenths of a degree; (2) a cathetometer for reading the thermometer (this may be done with an eyeglass if held steadily and properly adjusted); (3) a thermometer; (4) a tall beaker glass 35 cm. high and 10 cm. in diameter; (5) a test tube 30 cm. long and 3.5 cm. in diameter; (6) a stand for supporting the apparatus; (7) some method of stirring the water in the beaker, for example, a blowing bulb of rubber, and a bent glass tube extending to near the bottom of the beaker; (8) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

Manipulation.—The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of 15 to 20 cm. on a smooth piece of ice floating in water. The disks thus formed are from 1 to 1.5 cm. in diameter, and weigh about 200 milligrams. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The mixture of alcohol and water is prepared by boiling distilled water and 95 per cent alcohol for ten minutes to remove the gases which they may hold in solution. While still hot the water is poured into the test tube already described until it is nearly half full. The test tube is then nearly filled with the hot alcohol. It should be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top.

The test tube containing the alcohol and water is placed in a tall beaker containing water and ice until cold. The disk of fat is then dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules,

The delicate thermometer is placed in the test tube, and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk, the thermometer is moved from time to time in a circularly pendulous manner.

The disk having been placed in position, the water in the beaker glass is slowly heated, and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° below the melting point, the disk of fat begins to shrivel, and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotatory movement should be given to the thermometer bulb. The rise of temperature should be so regulated that the last 2 degrees of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so, the reading of the thermometer is to be made. As soon as the temperature is taken, the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test tube (ice water having been used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum of about 1.5° above the melting point of the fat under examination.

The distilled water for floating the piece of ice on which the disks are made should be recently boiled, to free it of all air particles.

The edge of the disks should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place, and the disk adhere to the sides of the tube, a new trial should be made.

Triplicate determinations should be made, and the second and third results should show a near agreement.

Example.

Melting point of sample of butter:	°C
1st trial	33.15
2d trial	33.05
3d trial	33.00

ESTIMATION OF VOLATILE ACIDS.

Reagents.

(1) *Caustic soda solution.*—100 grams of NaOH are dissolved in 100 cc. of pure water. The caustic soda should be as free as possible from carbonates, and be preserved from contact with the air.

(2) *Alcohol*, of about 95 per cent, redistilled with caustic soda.

(3) *Acid.* Solution of sulphuric acid containing 25 cc. of strongest H_2SO_4 in 1,000 cc. of water.

(4) *Barium hydrate.*—An accurately standardized, approximately decinormal solution of barium hydrate.

(5) *Indicator.*—Alcoholic solution of phenol-phthalein.

Apparatus.

(1) *Saponification flasks* 250 to 300 cc. capacity of hard, well-annealed glass capable of resisting the tension of alcohol vapor at 100°.

(2) *A pipette* graduated to deliver 40 cc.

(3) *Distilling apparatus.*

(4) *Burette.*—An accurately calibrated burette reading to tenths of a cc.

Manipulation.

Weighing the fat.—The butter or fat to be examined should be melted, and kept in a dry warm place at about 60° for two or three hours, until the moisture and curd have entirely settled out. The clean supernatant fat is poured off and filtered through a dry filter paper in a jacketed funnel containing boiling water. Should the filtered fat in a fused state not be perfectly clear, the treatment above mentioned must be repeated.

The saponification flasks are prepared by having them thoroughly washed with water, alcohol, and ether, wiped pefectly dry on the outside, and heated for one hour to boiling temperature. The flasks should then be placed in a tray by the side of the balance and covered with a silk handkerchief until they are perfectly cool. They must not be wiped with a silk handkerchief within fifteen or twenty minutes of the time they are weighed. The weight of the flasks having been accurately determined, they are charged with the melted fat in the following way:

A pipette with a long stem, marked to deliver 5.75 cc. is warmed to a temperature of about 50°. The fat having been poured back and forth once or twice into a dry beaker in order to thoroughly mix it, is taken up in the pipette and the nozzle of the pipette carried to near the bottom of the flask, having been previously wiped to remove any adhering fat, and 5.75 cc. of fat are allowed to flow into the flask. After the flasks have been charged in this way, they should be re-covered with the silk handkerchief and allowed to stand fifteen or twenty minutes, when they are again weighed.

The saponification.—Ten cc. of 95 per cent alcohol are added to the fat in the flask, and then 2 cc. of the concentrated soda solution; a soft cork stopper is now inserted in the flask and tied down with a piece of twine. The saponification is then completed by placing the flask upon the water or steam bath. The flask during the saponification, which should last one hour, should be gently rotated from time to time, being careful not to project the soap for any distance up its sides. At the end of an hour the flask, after having been cooled to near the room temperature, is opened.

Removal of the alcohol.—The stoppers having been laid loosely in the mouth of the flask, the alcohol is removed by dipping the flask into a steam bath. The steam should cover the whole of the flask except the neck. After the alcohol is nearly removed, frothing may be noticed in the soap, and to avoid any loss from this cause or any creeping of the soap up the sides of the flask, it should be removed from the bath and shaken to and fro until the frothing disappears. The last traces of alcohol vapor may be removed from the flask by waving it briskly, mouth down, to and fro.

Dissolving the soap.—After the removal of the alcohol the soap should be dissolved by adding 100 cc. of recently boiled distilled water, warming on the steam bath with occasional shaking, until solution of the soap is complete.

Setting free the fatty acids.—When the soap solution has cooled to about 60° or 70° the fatty acids are separated by adding 40 cc. of the dilute sulphuric acid solution mentioned above.

Melting the fatty acid emulsion.—The flask should now be re-stoppered as in the first instance, and the fatty acid emulsion melted by replacing the flask on the steam bath. According to the nature of the fat examined, the time required for the fusion of the fatty acid emulsions may vary from a few minutes to several hours.

The distillation.—After the fatty acids are completely melted, which can be determined by their forming a transparent oily layer on the surface of the water, the flask is cooled to room temperature, and a few pieces of pumice stone added. The pumice stone is prepared by throwing it, at a white heat, into distilled water, and keeping it under water until used. The flask is now connected with a glass condenser slowly heated with a naked flame until ebullition begins, and then the distillation continued by regulating the flame in such a way as to collect 110 cc. of the distillate in, as nearly as possible, thirty minutes. The distillate should be received in a flask accurately graduated at 110 cc.

Titration of the volatile acids.—The 110 cc. of distillate, after thorough mixing, are filtered through perfectly dry filter paper and collected in a flask graduated at 100 cc. The 100 cc. of the filtered distillate are poured into a beaker holding from 200 to 250 cc., 0.5 cc. phenolphthalein solution added, and decinormal barium hydrate run in until a red color is produced. The contents of the beaker are then returned to the measuring flask to remove any acid remaining therein, poured again into the beaker, and the titration continued until the red color produced remains apparently unchanged for two or three minutes. The number of cubic centimeters of decinormal $Ba(OH)_2$ required should be increased by one-tenth.

ALTERNATE METHOD OF DETERMINING VOLATILE ACIDS.

Saponification without the use of alcohol.—To avoid the danger of loss from the formation of ethers, and the trouble of removing the alcohol after saponification, the fat may be saponified with a solution of caustic potash in a closed flask without using alcohol. The operation is carried on exactly as indicated above for saponification in a closed flask, using caustic potash solution instead of soda, and omitting the operation for volatilizing the alcohol. The caustic potash is prepared as follows: Dissolve 100 grams of the purest potassium hydrate in 58 grams of hot distilled water. Allow to cool in a stoppered vessel, decant the clear solution, and preserve in a vessel out of contact with the air. For the saponification use 2 cc. of the caustic-potash solution, which are poured on the fat after it has solidified in the flask. Great care must be taken that none of the fat is allowed to rise on the sides of the saponifying flask to a point where it can not be reached by the alkali. During the process of saponification the flask can only be very gently rotated in order to avoid the difficulty mentioned. This process is not recommended in any except a closed flask with round bottom. In the subsequent solution of the soap use only 80 cc. of distilled water, and in setting free the fatty acids use 60 cc. of the dilute sulphuric acid. In other respects the distillation is conducted as previously described. Potash is used instead of soda, so as to form a softer soap, and thus allow a more perfect saponification.

The saponification may also be conducted as follows: The alkali and fat in the melted state are shaken vigorously in the saponification flask until a complete emulsion is secured. The rest of the operation is then conducted as above.

IODIN ABSORPTION NUMBER.

Reagents.

(*1*) *Iodin solution.*—Dissolve 25 grams of pure iodin in 500 cc. of 95 per cent alcohol. Dissolve 30 grams of mercuric chlorid in 500 cc. of 95 per cent alcohol. The last solution, if necessary, is filtered, and then the two solutions mixed. The mixed solution should be allowed to stand twelve hours before using.

(*2*) *Decinormal sodium thiosulphate solution.*—Take 24.6 grams of chemically pure sodium thiosulphate freshly pulverized as finely as possible and dried between filter or blotting paper. Make this up to 1,000 cc. at the temperature at which the titrations are to be made.

(*3*) *Starch paste.*—One gram of starch boiled in 200 cc. of distilled water for ten minutes and cooled to room temperature.

(*4*) *Solution of potassium iodid.*—One hundred and fifty grams of potassium iodid dissolved in water and made up to 1 liter.

(*5*) *Solution of potassium bichromate.*—Dissolve 3.874 grams of chemically pure potassium bichromate in distilled water and make the volume up to 1 liter at the temperature at which the titrations are to be made.

Manipulation.

Standardizing the sodium thiosulphate solution.—Run 20 cc. of the potassium bichromate solution, to which has been added 10 cc. of the solution of potassium iodid, into a glass-stoppered flask. Add to this 5 cc. of strong hydrochloric acid. Allow the solution of sodium thiosulphate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste and with constant shaking continue to add the sodium thiosulphate solution until the blue color just disappears. The number of cubic centimeters of thiosulphate solution used multiplied by 5 is equivalent to 1 gram of iodin.

Example.—Twenty cc. $K_2Cr_2O_7$ solution required 16.2 cc. sodium thiosulphate; then $16.2 \times 5 = 81$ = number cubic centimeters of thiosulphate solution equivalent to 1 gram of iodin. Then 1 cc. thiosulphate solution = 0.0124 gram of iodin. Theory for decinormal solution of sodium thiosulphate, 1 cc. = 0.0127 gram of iodin.

Weighing the sample.—About 1 gram of butter fat is to be weighed in a glass-stoppered flask holding about 300 cc., with the precautions mentioned for weighing the fat for determining volatile acids.

Absorption of iodin.—The fat in the flask is dissolved in 10 cc. of chloroform. After complete solution has taken place, 30 cc. of the iodin-mercuric chlorid solution is added. The flask is now placed in a dark place and allowed to stand, with occasional shaking, for three hours.

Titration of the unabsorbed iodin.—100 cc. of distilled water are added to the contents of the flask, together with 20 cc. of the potassium iodid solution. Any iodin which may be noticed upon the stopper of the flask should be washed back into the flask with the potassium iodid solution. The excess of iodin is now taken up with the sodium thiosulphate solution, which is run in gradually, with constant shaking, until the yellow color of the solution has almost disappeared. A few drops of starch paste are then added, and the titration continued until the blue color has entirely disappeared. Toward the end of the reaction the flask should be stoppered and violently shaken, so that any iodin remaining in solution in the chloroform may be taken up by the potassium iodid solution in the water. A sufficient quantity of sodium thiosulphate solution should be added to prevent a reappearance of any blue color in the flask for five minutes.

Setting the value of the iodin solution by the thiosulphate solution.—At the time of adding the iodin solution to the fats, two blank flasks of the same size as those used for the determination should be employed for conducting the operation described above, but without the presence of any fat. In every other respect the performance of the blank experiments should be just as described. These blank experiments must be made each time the iodin solution is used.

Example—Blank determinations.

(1) 30 cc. iodin solution required 46.4 cc. sodium thiosulphate solution.
(2) 30 cc. iodin solution required 46.8 cc. of sodium thiosulphate solution.
Mean, 46.6.

Per cent of iodin absorbed.

Weight of fat taken	grams..	1.0479
Quantity of iodin solution used	cubic centimeters..	0.30
Thiosulphate equivalent to iodin used	do....	46.6
Thiosulphate equivalent to remaining iodin	do....	14.7
Thiosulphate equivalent to iodin absorbed	do....	31.9

Per cent of iodin absorbed, $31.9 \times 0.0124 \times 100 \div 1.0479 = 37.75$.

ELECTIVE METHODS OF BUTTER ANALYSIS.

The elective methods of butter analysis remain as described in Bulletin No. 31, pages 200–202.

METHODS FOR MILK ANALYSIS.

DETERMINATION OF WATER.

Evaporate 1 to 2 grams of milk in a tared flat dish containing from 15 to 20 grams of pure dry sand, or without sand, until apparently dry. Dry and heat for one hour at the temperature of boiling water. Cool in a desiccator, and weigh rapidly to avoid absorption of hygroscopic moisture.

DETERMINATION OF TOTAL NITROGENOUS MATTER.

Place in a Kjeldahl digestion flask a known weight (about 5 grams) of milk, and proceed, without evaporation, exactly as described for this method under nitrogen.

DETERMINATION OF FAT—PAPER-COIL METHOD.

Coils made of thick filter paper, cut into strips 6.25 by 62.5 mm. are thoroughly extracted with ether and alcohol, or the weight of the extract corrected by a constant obtained for the paper. If this latter method is used a small amount of anhydrous sodium carbonate should be added. From a weighing bottle about 5 grams of milk are transferred to the coil by a pipette, care being taken to keep the end of the coil held in the fingers dry. The coil, dry end down, on a piece of glass, is dried at the temperature of boiling water for one hour, or, better, dried in hydrogen at the temperature of boiling water, transferred to an extraction apparatus, and extracted with absolute ether or petroleum spirit boiling at about 45°. The extracted fat is dried in hydrogen and weighed.

If the milk is sour add about 10 per cent, by weight, of strong ammonia water, and a small quantity of anhydrous sodium carbonate, correcting the results for the ammonia added.

ALTERNATE METHOD FOR DETERMINING WATER AND FAT IN MILK.

In the bottom of a perforated test tube is placed a plug of clean cotton, the tube is then filled three-fourths full of ignited asbestos, lightly packed, and a plug of cotton inserted over it. The tube and contents are weighed, the plug of cotton carefully removed, 5 grams of milk from a weighed pipette run into it, and the plug of cotton replaced. The tube, connected at its lower end by a rubber tube and adapter with a filter pump, is placed in a drying oven at a temperature of boiling water, and a slow current of dry air drawn through it until dry.

The tube containing the solids from the above operation is placed in an extraction apparatus and exhausted with ether in the usual way.

DETERMINATION OF SUGAR.

Reagents.

(1) *Basic lead acetate,* sp. gr. 1.97.—Boil a saturated solution of sugar of lead with an excess of litharge and make it of the strength indicated above. 1 cc. of this will precipitate the albumens in 50 to 60 cc. of milk.

(2) *Acid mercuric nitrate.*—Dissolve mercury in double its weight of nitric acid, sp. gr. 1.42. Add to the solution an equal volume of water. 1 cc. of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarization.

(3) *Mercuric iodid with acetic acid.*—KI, 33.2 grams, $HgCl_2$, 13.5 grams, $C_2H_4O_2$, 20 cc. H_2O, 64 cc.

Apparatus.

(1) Pipettes marked at 59.5, 60, and 60.5 cc. (2) Sugar flasks marked at 102.4 cc. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

Manipulation.—(1) The room and milk should be kept at a constant temperature.

(2) The specific gravity of the milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required use specific gravity flasks

(3) If the specific gravity be 1.026, or nearly so, place 60.5 cc. in the sugar flask. Add 1 cc. of mercuric nitrate solution, or 30 cc. of mercuric iodid solution, and fill to the 102.4 cc. mark. The precipitated albumen occupies a volume of about 2.44 cc. Hence the milk solution is really 100 cc. If the specific gravity is 1.030, use 60 cc. of milk. If the specific gravity is 1.034, use 59.5 cc. of milk.

(4) Fill up to the mark in the 102.4 cc. flask, shake well, filter, and polarize.

NOTES.—In the above method of analysis the specific rotatory power of milk sugar is taken at 52.5, and the weight of it in 100 cc. solution to read 100 degrees on the cane-sugar scale at 20.56 grams. This is for instruments requiring 16.19 grams sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk sugar, whatever instrument is employed.

Since the quantity of milk taken is three times 20.56 grams, the polariscopic readings divi ed by 3 give at once the percentage of milk sugar when a 200-mm. tube is used.

If a 400-mm. tube is employed, divide the reading by 6; if a 500-mm. tube is used, divide by 7.5.

By using a flask graduated at 102.4 cc. for 60 cc. no correction for volume of precipitated casein need be made. In no case is it necessary to heat the sample before polarizing.

ALTERNATE METHOD FOR SUGAR.

The sugar may also be determined, either gravimetrically or volumetrically, by alkaline copper solution, as described under sugar analysis.

ESTIMATION OF ASH.

In a weighed dish put 20 cc. of milk from a weighing bottle; add 6 cc. of HNO_3, evaporate to dryness, and burn at low red heat until ash is free from carbon.

PROVISIONAL METHODS OF CHEESE ANALYSIS.

SAMPLING.

Where the cheese can be cut, a narrow wedge reaching from the edge to the center of the cheese will more nearly represent the average composition of the cheese than any other sample. This may be chopped quite fine, with care to avoid evaporation of water, and the several portions for analysis taken from the mixed mass. When the sample is taken with a cheese tryer, a plug taken perpendicular to the surface one-third of the distance from the edge to the center of the cheese should more nearly represent the average composition than any other. The plug should either reach entirely through or only half way through the cheese. For inspection purposes the rind may be rejected, but for investigations, where the absolute quantity of fat in the cheese is required, the rind should be included in the sample. It is well, when admissible, to take two or three plugs on different sides of the cheese and, after splitting them lengthwise with a sharp knife, take portions of each for the different determinations.

DETERMINATION OF WATER.

From 5 to 10 grams of cheese should be taken, and placed in thin slices in a weighed platinum or porcelain dish which contains a small quantity of freshly ignited asbestos, to absorb the fat which may run out of the cheese. This is then heated in a boiling water oven for ten hours and weighed; the loss in weight shall be considered as water. Or if preferred the dish may be placed in a desiccator over concen-

trated sulphuric acid and dried to constant weight. In some cases this may require as much as two months. The acid should be renewed when the cheese has become nearly dry.

DETERMINATION OF ASH.

The dry residue from the water determination may be taken for the ash. If the cheese be rich the asbestos will be saturated therewith. This may be ignited carefully and the fat allowed to burn off, the asbestos acting as a wick. No extra heat should be applied during this operation, as there is danger of spurting. When the flame has died out the burning may be completed in a muffle at low redness. When desired, the salt may be determined in the ash in the manner specified for butter.

DETERMINATION OF FAT.

Take 5 to 10 grams of cheese and grind it in a small mortar with about twice its weight of anhydrous copper sulphate. The grinding should continue until the cheese is finely pulverized and evenly distributed throughout the mass, which will have a uniform light blue color. This mixture is transferred to a glass tube which has a strong filter paper, supported by a piece of muslin, tied over one end. Put a little of the clean anhydrous copper sulphate into the tube next to the filter before introducing the mixture containing the cheese. On top of the mixture place a tuft of ignited asbestos, and place the tube in a continuous extraction apparatus and extract with anhydrous ether for fifteen hours. Dry the fat obtained at 100° to constant weight.

DETERMINATION OF CASEIN.

Make a determination of nitrogen by the Kjeldahl method, taking about 2 grams of cheese, and multiply the result by 6.25 for casein.

OTHER CONSTITUENTS.

The sum of the percentages of the different constituents, determined as above, subtracted from 100 will give the amount of organic acids, milk sugar, etc., in the cheese.

SOIL AND ASH ANALYSIS.—PROVISIONAL METHODS FOR THE YEARS 1892 AND 1893.

SOIL.

SAMPLING.

The soil selected should be, as far as possible, in its natural condition, not modified by recent applications of manure, or changed by the transporting action of water or wind. Surface accumulations of decaying leaves, etc., should be removed before taking the sample. To eliminate accidental variations in the soil, select specimens from five or six places in the field which seem to be fair averages of the soil, remove 2 or 3 pounds of the soil, taking it down to the depth of 6 to 9 inches, unless there is a change from top soil to subsoil at a less depth, so as to include the whole depth of the soil. Mix these soils intimately, remove any stones, shake out all roots and foreign matters, and dry the soil until it becomes friable. Break down any lumps in a mortar with wooden pestle, but avoid pulverizing any mineral fragments; pass 8 to 10 pounds of the soil through a 1 mm. sieve, rejecting all pebbles and material too coarse to pass through the sieve, noting the percentage amount of soil so rejected. If the amount equals or exceeds 20 per cent this should be pulverized to pass through a 2 mm. sieve, and in this state submitted to analysis. Once

more mix intimately the sifted soil. Expose in thin layers in a warm room till thoroughly air-dry (or dry it in air bath at temperature of 40°), place 6 to 8 pounds in a clean bottle, with label of locality and date, and cork tightly.

The soil is rapidly dried to arrest nitrification; it is not heated above 40° lest there be dissipation of ammonia compounds, or a change in the solubility of the soil. The normal limit to which the soil may be heated in place by the sun's rays should not be exceeded in preparing a soil for an agricultural chemical analysis.

HYGROSCOPIC MOISTURE.

Place 5 grams of air-dry soil in a flat-bottomed and tared platinum dish; heat in air bath to 110° for eight hours; cool in a desiccator, and weigh; repeat the heating, cooling, and weighing at intervals of an hour till constant weight is found, and estimate the hygroscopic moisture by the loss of weight. Weigh rapidly to avoid absorption of moisture from the air.

WATER-HOLDING POWER.

In the throat of a clean 3-inch glass funnel place a very small filter, just large enough to prevent the soil from running through the stem; wet the filter and add 100 grams of the air-dry soil; from a graduate containing 100 cc. pour water on the soil till it is thoroughly wet and a few drops pass through; let it stand undisturbed till no more water flows from the soil, the funnel being covered with a glass plate to prevent evaporation. Return the water which has filtered through to the graduate. The number of cc. taken up by the soil will show the percentage capacity of the soil to hold water.

In using this process certain precautions are necessary to secure uniform results. The soil should be simply poured into the funnel, and not pressed or compacted in any way. When water is poured upon the soil, no disturbing influence, no handling or shaking of the soil, should take place.

CAPILLARY POWER OF SOILS.

This is determined by filling a long glass tube with soil, placing the lower end in water, and marking the height to which the water ascends, as shown by the changed color of the soil in the tube.

VOLATILE MATTER.

The platinum crucible and 5 grams of soil used to determine the hygroscopic moisture may be used to determine the volatile matter. Heat the crucible and dry soil to low redness. The heating should be prolonged till all organic material is burned away, but below the temperature at which alkaline chlorids volatilize. Moisten the cold mass with a few drops of a saturated solution of ammonium carbonate, dry, and heat to 150° to expel excess of ammonia. The loss in weight of the dry soil represents organic matter, water of combination, salts of ammonia, etc.

WATER-SOLUBLE MATERIALS OF THE SOIL.

To prepare a water extract of the soil, a percolator of glass or tin may be employed. It should be large enough to hold one kilo of soil.

By means of a perforated rubber stopper connect the neck of the percolator with a two-necked bottle of 2 liters capacity, pour sufficient distilled water (ammonia free) on the soil to moisten it all, and let the whole stand undisturbed for half an hour, then add more pure distilled water, and if the filtration is too slow use the filter pump, till a liter of filtrate is secured. If the soil extract is cloudy, filter through a plain filter.

(1) *Soluble solids.*—Evaporate 100 cc. to dryness on the water bath in a tared

dish to determine the percentage of water-soluble materials in the soil; each gram of residue representing 1 per cent of such materials. Test this dry residue for nitrates by pouring over it 10 cc. of pure H_2SO_4, holding in solution 3 or 4 milligrams of brucin sulphate.

(2) *Chlorids.*—Titrate 100 cc. with standard decinormal silver nitrate with two drops of a solution of K_2CrO_4 as an indicator. Titrate in a white porcelain dish and view the reaction through a yellow glass plate of such tint as will eliminate the color of the chromic solution. The reaction will then be sharply defined.

(3) *Sulphates.*—Precipitate the sulphates in 100 cc. of the soil extract with $BaCl_2$, in presence of a few drops of HCl.

Reserve the rest of the water solution for the estimation of nitrates.

ACID-SOLUBLE MATERIALS.

In the following scheme for soil analysis it is recommended to use the air-dry soil from the sample bottle for each separate investigation. A determination made once for all of hygroscopic moisture and of water of combination on a separate specimen of air-dry soil will afford corrections for all the other samples used. It is not desirable to ignite the soil before analysis or to heat it so as to change its chemical properties.

(1) *Acid digestion of the soil.*—Place 10 grams of the air-dry soil in a 150 to 200 cc. Bohemian glass flask, add 100 cc. of pure HCl of sp. gr. 1.115, insert the stopper, wire it securely, place in a steam bath, and digest for thirty-six hours at the temperature of boiling water. Pour the contents of the flask into a small beaker, wash out with distilled water, add the washings to the contents of the beaker, and filter through a washed filter. Residue is the amount insoluble in hydrochloric acid. Add a few drops of HNO_3 to the filtrate, and evaporate to dryness on the water bath; take up with hot water and a few drops of HCl, and again evaporate to complete dryness. Take up as before, and filter into a liter flask, washing with hot water. Cool and make up to mark. This is solution "A." The residue is soluble silica.

(2) *Ferric oxid, alumina, and phosphoric acid.*—To 100 cc. or 200 cc., according to the probable amount of iron present, of the solution A, add NH_4OH to alkaline reaction (avoiding excess), to precipitate ferric and aluminic oxids and phosphates. Expel the excess of ammonia by boiling, allow to settle, decant the clear solution through a filter; add to the flask 50 cc. of hot distilled water, boil, settle, and decant as before. After pouring off all the clear solution possible dissolve the residue with a few drops of HCl with heat, and add just enough NH_4HO to precipitate the oxids. Wash by decantation with 50 cc. of distilled water, and then transfer all the precipitate to the filter and wash with hot distilled water till the filtrate becomes free from chlorids. Save the filtrate and washings which form solution B. Dry the filter and precipitate at 110°, transfer the precipitate to a tared platinum crucible, burn the filter and add the ash to the precipitate, heat the whole red hot, cool in desiccator, and weigh. The increase of weight, minus the ash of filter and the phosphoric acid (found in a separate process), represents the weight of the ferric and aluminium oxids.

(3) *Ferric oxid.*—Precipitate 100 cc. of solution A, as under 2, except that only one precipitation is made; wash with hot water; dissolve while wet in dilute H_2SO_4; reduce with zinc and estimate ferric oxid by a standard solution of potassium permanganate. To prepare the potassium permanganate solution, dissolve 3.156 grams of pure crystallized potassium permanganate in 1,000 cc. of distilled water, and preserve in a ground-glass-stoppered bottle, shielded from the light. Standardize this solution with pure ferrous sulphate or ammonio-ferrous sulphate or oxalic acid.

The weight of ferric oxid deducted from ferric oxid and alumina (12), with corrections for filter ash and phosphoric acid, will give the weight of alumina in 2 grams of air dry soil.

(*4*) *Phosphoric acid.*—This may be estimated in the above iron solution if the soil is sufficiently rich, by the molybdate method, given under fertilizers; or if the quantity of soil represented in the iron solution is not sufficient, a fresh portion of solution A may be taken, and the phosphoric acid determined directly by the molybdate method.

(*5*) *Manganese.*—Concentrate the filtrate and washings (solution B) to 200 cc. or less; add NH_4OH to alkalinity; add bromin water and heat to boiling, keeping beaker covered with watch crystal; as the bromin escapes the beaker is allowed to cool somewhat, ammonia and bromin water again added, and heated as before.

This process is continued until the manganese is completely precipitated, which requires from thirty to sixty minutes, and the solution filtered while still warm, the precipitate washed, dried, ignited, and weighed; estimate as Mn_3O_4.

(*6*) *Lime.*—If no manganese is precipitated, add to solution B, or the filtrate and washings from (5) 20 cc. of a strong solution of NH_4Cl and 40 cc. of saturated solution of $(NH_4)_2C_2O_4$ to completely precipitate all the lime as oxalate and convert the magnesia into soluble magnesium oxalate. Heat to boiling and let stand for six hours till the calcium oxalate settles clear, decant the clear solution on a filter, pour 50 cc. of hot distilled water on the precipitate and again decant the clear solution on the filter, transfer the precipitate to the filter, and wash it free from all traces of oxalates and chlorids. Dry, and ignite the precipitate over the blast lamp until it ceases to lose weight, weigh and estimate as CaO; carefully moisten with H_2SO_4, heat the inclined covered crucible gently to avoid loss, then intensely, and weigh as $CaSO_4$

(*7*) *Magnesia.*—Concentrate the filtrate and washings (from 6) to 200 cc., place in a half-liter Erlenmeyer flask, add 30 cc. of a saturated solution of Na_2HPO_4 and 20 cc. of concentrated NH_4HO, cork the flask, and shake violently at intervals of a few minutes till crystals form, then set the flask in a cool place for twelve hours. Filter off the clear liquid through a tared Gooch filter, transfer the precipitate to the filter, and wash with dilute ammonium hydrate (1 : 3) till the filtrate is free from phosphates; dry and ignite the crucible, at first gently and then intensely, to form magnesium pyrophosphate. The increase of weight $\times$ 0.36024 = MgO. By using an Erlenmeyer flask free from scratches and marks, and shaking violently instead of stirring with a glass rod, the danger is almost entirely avoided of crystals adhering to the sides of the vessel; but if crystals do adhere they are readily removed by a rubber-tipped glass rod.

(*8*) *Sulphuric acid.*—Evaporate 200 cc. of solution A (1) nearly to dryness on a water bath to expel the excess of acid; then add 100 cc. of distilled water; heat to boiling and add 10 cc. of a solution of $BaCl_2$, and continue the boiling for five minutes. When the precipitate has settled pour the clear liquid on a tared Gooch filter, treat the precipitate with 50 cc. of boiling water, and transfer the precipitate to the filter and wash with boiling water till the filtrate is free from chlorids. Dry the filter and ignite strongly. The increase in weight is barium sulphate, which multiplied by 0.34331 = SO_3 in 2 grams of air-dry soil.

(*9*) *Potash and soda.*—[To another portion of 200 cc. of solution add $BaCl_2$ in slight excess, and make alkaline with ammonia to precipitate sulphuric and phosphoric acids, ferric oxid, etc. Then precipitate the calcium and barium by ammonium oxalate.[1]] Evaporate the filtrate and washings to dryness, heat to a low red heat to decompose oxalates and expel ammonia salts, dissolve in 25 cc. of distilled water, filter and wash the precipitate, add to the filtrate and washings 10 cc. of baryta water, and digest for an hour. Filter and wash the precipitate, add ammonium carbonate to the filtrate to complete precipitation of baryta, filter and wash this precipitate. Evaporate the filtrate and washings in a tared platinum dish, gently ignite the residue to expel ammonia salts, cool and weigh. The increase of weight represents the potassium and sodium chlorids in 2 grams of air-dry soil.

[1] NOTE BY THE SECRETARY.—The sentences between brackets were inserted to repair an evident omission.

Separate and estimate the potassium chlorid by platinic chlorid according to the method given for potash determination.

Subtract the weight of potassium chlorid as thus found from the weight of potassium chlorid and sodium chlorid. The difference represents sodium chlorid.

Alternate method.—For an alternate method for alkalis, use J. Lawrence Smith's method as given in Crookes' Select Methods, second edition, pp. 28 to 40.

(*10*) *Other alkali metals.*—The salts of lithium, cesium, and rubidium are occasionally found in very small amounts in soils. The agricultural uses of these salts are still in question, and their amount is too small to admit of quantitative estimation. A quantitative examination may be made by the spectroscope with the water-soluble materials evaporated to dryness and dissolved with 2 or 3 drops of HCl. Test by the spectroscope with a platinum wire in a Bunsen flame.

NITROGEN OF THE SOIL.

The nitrogen compounds in the soil are usually placed in three classes:

1. The nitrogen combined with oxygen as nitrates or nitrites, existing as soluble salts in the soil.

2. The nitrogen combined with hydrogen as ammonia, or organic nitrogen easily convertible into ammonia. The ammonia may exist as salts, or be occluded by hydrated ferric or aluminum oxids and organic matter in the soil.

3. The inert nitrogen of the soil or the humus nitrogen.

Active soil nitrogen.—The material proposed for reducing the nitrates to ammonia, and at the same time to bring ammonia salts and organic nitrogen into a condition for separation by distillation, is sodium amalgam. Liquid sodium amalgam may be readily prepared by placing 100 cc. of mercury in a flask of half a liter capacity, covering the warmed mercury with melted paraffin and dropping into the flask at short intervals pieces of metallic sodium the size of a large pea (taking care that the violence of the reaction does not project the contents from the flask), till 6.75 grams of sodium have combined with the mercury. The amalgam contains 0.5 per cent of sodium and may be preserved indefinitely under the covering of paraffin.

To estimate the active soil nitrogen, weigh 50 grams of air-dry soil and place it in a clean mortar. Take 200 cc. of ammonia-free distilled water, rub up the soil with a part of the water to a smooth paste, transfer this to a flask of 1 liter capacity, washing the last traces of the soil into the flask with the rest of the water. Add 25 cc. of the liquid sodium amalgam and shake the flask so as to break the sodium amalgam into small globules distributed through the soil. Insert a stopper with a valve, and set aside in a cool place for twenty-four hours. Pour into the flask 50 cc. of milk of lime and distill, on a sand bath, 100 cc. into a flask containing 20 cc. of decinormal sulphuric acid, and titrate with decinormal soda solution, using dimethylorange as indicator. Estimate the nitrogen of the ammonia found as active soil nitrogen.

If the ammonia produced is too small in amount to be readily estimated volumetrically, determine the ammonia by Nesslerizing the distillate.

Estimation of nitrates in the soil.—When it is desired to estimate separately the nitrates in the soil the following method may be used: Evaporate 100 cc. of the soil extract to dryness on the water bath; dissolve the soluble portion of the residue in 100 cc. of ammonia-free distilled water, filtering out any insoluble residue, place the solution in a flask, and add 10 cc. of liquid sodium amalgam, insert stopper with valve, set it aside to digest in a cool place for twenty-four hours, add 50 cc. of milk of lime, distil and titrate as in 22, and estimate the nitrogen as N_2O_5.

Nesslerizing may be substituted for titration when the amount of nitrates is small.

An approximate estimation of the amount of nitrates will be of value in determining which method of estimation to use. This may be done by evaporating a measured quantity of the soil extract say 5 cc., on a porcelain crucible cover on a steam bath or radiator, having first dissolved a minute fragment of pure brucin

sulphate in the soil extract. When dry pour over the residue concentrated sulphuric acid, free from nitrates, and observe the color reactions produced.

If the nitrate (reckoned as KNO_3) left upon evaporating the quantity of water taken does not exceed the two-thousandth part of a milligram, only a pink color will be developed by adding the sulphuric acid; with the three-thousandth part of a milligram, a pink with faint reddish lines; with the four-thousandth part, a reddish color; with the five-thousandth part, a red color.

By increasing or diminishing the amount of soil extract evaporated to secure a color reaction of a certain intensity, an approximate estimate may be made of the amount of nitrates present.

Blank experiments to test the acid and the brucin sulphate will be required before confidence can be placed in such estimations.

Total nitrogen of soils.—The total nitrogen of soils may be determined by the usual combustion with soda-lime, but this process is often unsatisfactory because of the large amount of material required when the organic matter or humus is small in amount.

A modification of the Kjeldahl method is more easy to carry out and gives results equally satisfactory. Place 20 grams of soil in a Kjeldahl flask, and add 20 cc. of sulphuric acid (free from ammonia) holding in solution 1 gram of salicylic acid. (If the soil contains much lime or magnesia in the form of carbonate, enough more sulphuric acid must be added to secure a strongly acid condition of the contents of the flask. Add gradually 2 grams of zinc dust, shaking the contents of the flask to secure intimate mixture. Place the flask in a sand bath and heat till the acid boils, and maintain the boiling for 10 minutes. Add 1 gram of mercury and continue the boiling for 1 hour, adding 10 cc. of sulphuric acid if the contents of the flask are likely to become solid. Cool the flask and wash out the soluble materials with 200 cc. of pure water, leaving the heavy earthy materials. Rinse the residue with 100 cc. of water, and add this to the first washings. Place this soluble acid extract in a liter digestion flask, add 35 cc. of a solution of potassium sulphid, and shake the flask to secure intimate mixture of the contents. Introduce a few fragments of granulated zinc, pour in 75 cc. of a saturated solution of caustic soda, connect the flask with a condenser, and distill 150 cc. into a flask containing 20 cc. of acid, using the same acid and alkali for titration used in Kjeldahl method under fertilizers.

Enter the nitrogen found in this operation as total soil nitrogen.

The difference between the total soil nitrogen and the active soil nitrogen will express the inert nitrogen of the soil.

CARBON DIOXID.

This is determined as under ash analysis—using 1 to 5 grams—according to the amount of CO_2 present.

ASH.

PREPARATION OF ASH.

Before combustion the material must be thoroughly cleaned from all foreign matter, especially from adhering soil, wood, bark, roots, etc.

The combustion should be carried on at a comparatively low temperature, never reaching a full red heat, because of the danger of volatilizing alkaline chlorids, etc., nor in a strong draft of air, lest the lighter parts of the ash be carried away.

Combustion is best carried on in a flat platinum dish in a muffle.

With substances rich in silica and alkalis it is better to first char the substance. Wash with distilled water to remove soluble salts, then dry and incinerate the residue. Evaporate the watery extract and add this to the rest of the ash.

With substances rich in phosphates, *e. g.*, seeds and animal substances, char the material and remove salts by acetic acid, decant the acetic solution, wash with distilled

water, and then complete the combustion. Add the acetic solution and washings to the final ash, evaporate to dryness, and gently ignite the whole to decompose the acetates. In whatever way obtained the whole of the ash should be pulverized and intimately mixed before analysis.

ANALYSIS OF WOOD ASHES.

Pass 100 grams of dry ashes through a wire sieve (of 1 mm. mesh) to separate materials manifestly foreign, *e. g.*, nails, broken glass and pottery, pebbles, etc., and estimate the per cent of such accidental materials. Pulverize any charcoal and semi-fused portions of ash remaining on the sieve, sift them and mix intimately with the sifted ashes, and preserve in stoppered bottles for analysis.

(*1*) *Moisture.*—Take 5 grams of the prepared sample in a tared platinum dish and heat to 110° in an air bath to constant weight.

(*2*) *Carbon.*—Heat this dried ash in a platinum dish till the ash is uniformly grayish white and there is no further loss of weight.

(*3*) *Sand and silica.*—Place 10 grams of ignited ash in a 150 cc. Bohemian flask, glass stoppered, measure out 100 cc. of HCl (specific gravity 1.115) and pour on the ash cautiously to prevent loss, and when all effervescence has ceased, add the rest of the acid; insert the glass stopper, wire it securely, and place in a steam bath for two hours; empty the flask into a platinum dish, wash with distilled water, adding the washings to the ash solution, and evaporate the whole to dryness on a water bath. Add 10 cc. of dilute HCl and 50 cc. of distilled water to the contents of the platinum dish, transfer the contents to a filter, wash with distilled water till the last drops of the filtrate are free from chlorids, dry and ignite the precipitate, and filter. If there are no grains of sand (revealed by grittiness when stirred with a glass rod), subtract the ash of the filter from the weight of this residue and estimate the rest as silica. If sand is present, boil the ignited and weighed residue in a strong solution of Na_2CO_3 to dissolve silica, wash by decantation to remove all sodium salts, dry, and weigh the sand. The difference between the weight of sand, and silica + sand, will give the weight of silica.

The filtrate and washings from sand and silica are made up to 1,000 cc. (solution C) and kept for analysis.

(*4*) *Phosphoric acid.*—Take 100 or 200 cc. of solution C and estimate P_2O_5 by the official method.

(*5*) *Carbonic acid.*—Heat 4 or 5 grams of ash in a muffle till all charcoal is consumed; cool in a desiccator, transfer 2 grams to a carbonic-acid apparatus. Estimate the CO_2 in any convenient form of apparatus.

Alternate method.—By Liebig's potash bulbs. The usual process of absorption by solution of KHO, weighing, etc.

(*6*) *Chlorin.*—Pour out the nitric-acid solution from the CO apparatus upon a filter, wash out the last traces of the solution, pass the soluble matters through the filter and wash the insoluble residue with water acidulated with HNO_3. To this filtrate add solution of $AgNO_3$ to complete precipitation of the chlorids, boil and set aside in a dark place until the precipitate has settled completely, filter through a Gooch crucible, and wash with hot water. Dry at 110°, and weigh the silver chlorid.

(*7*) *Alternate method for chlorin.*—Boil 10 grams of ash in 400 cc. of pure water for half an hour, transfer all to a measuring flask of 500 cc. capacity, wash the beaker, and add the washings to the flask, cool, make up the volume to 500 cc. and mix intimately; filter off through a dry filter 100 cc., add a drop of solution of phenolpthalein, and neutralize with dilute HNO_3 till only a faint pink color remains, add 2 drops of a strong solution of K_2CrO_4 and titrate with standard decinormal solution of $AgNO_3$ (16.956 grams $AgNO_3$ to 1,000 cc.). Every cubic centimeter of the standard silver solution equals 0.003546 gram of chlorin.

The reliability of this method will depend upon the accuracy with which neutralization by nitric acid has been made. The least trace of free acid or alkaline carbonate will vitiate the results.

(*8*) *Sulphuric acid.*—Take an aliquot part of solution A and determine as under soils.

(*9*) *Oxid of iron.*—Take 200 cc. of the original acid solution, nearly neutralize with ammonia, then add 1 gram of sodium acetate, and acetic acid in slight excess, boil to precipitate ferric phosphate, filter while hot, and wash the precipitate with boiling distilled water till the filtrate is free from chlorids. Dissolve the precipitate on the filter with dilute H_2SO_4 into a small Erlenmeyer flask, wash the filter, dry and ignite and add the ashes to the acid solution in the flask, reduce the ferric to ferrous salt by zinc or by a coil of magnesium wire till a drop of the solution gives no color with NH_4CNS. Pour off the solution of ferrous salt into a beaker, rinse the flask and add the rinsings to the beaker, add freshly-boiled distilled water to make 200 cc. of the solution, add 2 cc. of sulphuric acid, heat to 70°, and titrate with standard solution of permanganate, and estimate the iron as ferric oxid.

(*10*) *Manganese.*—If a qualitative test shows the presence of manganese, evaporate the filtrate and washings from iron determination to 200 cc. or less, and estimate as under soils.

(*11*) *Lime.*—Evaporate the filtrate and washings from ferric phosphate to 100 cc. To the hot solution add 20 cc. of concentrated solution of ammonium chlorid and 40 cc. of saturated solution of ammonium oxalate; boil the whole for ten minutes and then let it stand in a warm place for six hours; decant the clear liquid upon a filter, wash the precipitate twice by decantation, then bring the precipitate upon the filter, and wash it free from chlorids and oxalates, testing the washings by silver nitrate. Ignite and estimate as under soils.

(*12*) *Magnesia.*—Evaporate the filtrate and washings from the calcium oxalate to 200 cc., pour into a clean and unscratched Erlenmeyer flask of 500 cc. capacity, add 30 cc. of strong solution of $(NH_4)_2HPO_4$ and 50 cc. of strong ammonium hydrate, cork the flask and shake violently at intervals of a few minutes till crystallization is established, and then set aside for twelve hours in a cold place. If crystals should form on the sides of the flask they are readily detached by a rubber-tipped rod, Filter through a tared Gooch filter, wash the precipitate with ammonium hydrate, diluted with distilled water (1 to 3), till the filtrate is free from phosphates. Dry the precipitate, ignite, at first very gently and then intensely, with blast lamp, to convert $Mg_2NH_4PO_4$ into $Mg_2P_2O_7$. Cool in desiccator and weigh. The increase of weight $\times$ .36024 = Mg O, in 2 grams of air-dry soil.

(*13*) *Estimation of alkalis.*—Concentrate the filtrate and washings from (8) to 100 cc, add NH_4HO and $(NH_4)_2C_2O_4$ to complete precipitation of barium and calcium, filter, wash the precipitate, evaporate the filtrate and washings to dryness in a platinum dish, and ignite gently. Add to the residue concentrated solution of oxalate of ammonia, evaporate to dryness, and ignite gently. Dissolve residue in distilled water, filter from insoluble MgO, acidify the filtrate with HCl, and evaporate to dryness in a tared platinum dish and ignite gently. The increase of weight represents the potassium and sodium chlorids. Separate and estimate potassium by $PtCl_4$ in the usual way, and determine the sodium by difference.

The ash of mineral coal contains only a small amount of alkalis and phosphates, but a large amount of insoluble material, clay, etc. The value depends mostly upon the sulphate of lime and phosphate present. They are often decomposed with difficulty. It should be ground to fine powder, and 5 grams placed in the digestion flask with 50 cc. of HCl (sp. gr. 1.115), and digested in the steam bath for six hours, and the soluble portion analyzed in the usual way.

METHODS OF ANALYSIS OF FERMENTED LIQUORS.

I. *Specific gravity.*—This determination is made with a picnometer, or a Westphal balance controlled by a picnometer, at a temperature of 15.5°.

II. *Alcohol.*—Weigh out about 100 grams of wine and distill in a proper distilling apparatus (provided with a glass condenser and mercury valve), after the addition

of 50 cubic centimeters of water. When about 100 cc. of distillate have come over the operation is stopped and the distillate weighed. Its specific gravity at 15.5° is then ascertained, by means either of a picnometer or a Westphal balance controlled by a picnometer, and the corresponding per cent of alcohol by weight obtained from the official tables. This figure, multiplied by the weight of distillate, and the result divided by the weight of sample taken gives the per cent of alcohol by weight contained in the sample.

III. *Extract.*—Fifty cc. (weighed), or in case of sweet wines a smaller amount, is evaporated on a water bath to a sirupy consistency in a platinum dish 85 millimeters in diameter and of about 75 cc. The residue is heated for two and one-half hours in a drying oven at 100°. In the case of sweet wines, that is, those containing more than 4 per cent of solids, not more than 10 cc. (weighed) should be taken.

Alternate method for extract in sweet wines.—Measure out carefully a definite volume of wine, transfer to an evaporating dish, and evaporate on a water bath until its volume is reduced to about one-third. Transfer the dealcoholized liquid to the graduated vessel in which it was originally measured out, rinse the dish carefully and make it up to the same volume with water, mix the liquid thoroughly, and ascertain its specific gravity, either by a picnometer or by an accurately standardized Balling or Brix spindle. The per cent of total solids, in case the picnometer is used, may be taken from the official tables herewith.

IV. *Acidity* (total acid constituents of the wine expressed as tartaric acid).—If carbonic acid is present, expel by shaking. Titrate with dilute alkali solution. The neutral point is determined by adding a drop of the solution to delicate litmus paper.

V. *Volatile acids* (expressed as acetic acid).—Distill in a current of steam, and titrate the carefully condensed distillate with standard alkali (decinormal).

VI. *Glycerol.*—(1) This is determined in dry wines as follows: The alcohol is driven off from 100 cc. of wine, lime or magnesia is added, and then evaporated to dryness. The residue is boiled with 90 per cent alcohol, filtered, and the filtrate evaporated to dryness. This residue is dissolved in 10 to 20 cc. of alcohol, 15 to 30 cc. of ether is added, and the mixture allowed to stand until it is clear. It is then decanted from the sticky precipitate into a glass-stoppered weighing bottle, evaporated to constant loss of weight, and weighed.

(2) The following method is employed for sweet wines: 100 cc. of wine is measured into a porcelain dish and evaporated on the water bath to a sirupy consistency, mixed with 100 to 150 cc. of absolute alcohol, poured into a flask, ether added in the proportion of one and one-half volumes to each volume of alcohol used, the flask well shaken, and allowed to stand until the liquid becomes clear. This is then poured off, and the residue again treated with a mixture of alcohol and ether. The liquids are mixed, the alcohol and ether driven off, the residue dissolved in water, and treated as in (1).

(3) It is necessary to test the glycerol from sweet wines for sugar, and if any is present it must be estimated by Soxhlet's method, and its weight subtracted from that of the glycerol.

VII. *Sugar.*—This is to be determined by Soxhlet's method. The presence of unfermented cane sugar is to be shown by inversion.

The wine should not contain more than 1 per cent of sugar.

Preliminary test.—100 cc. of wine accurately neutralized with normal soda solution is dealcoholized by evaporation to two-thirds, and the original volume restored with water (red wines are decolorized with pure animal charcoal); 25 cc. of copper solution and 25 cc. of Seignette salt solution are heated to boiling in a deep porcelain evaporating dish; wine is added from a burette till the fluid, after two minutes' boiling, no longer has a blue color. This gives an approximate estimate of the sugar present, and from this we can dilute the wine to very nearly 1 per cent of sugar.

Final determination.—25 cc. of wine, as in preliminary test, 25 cc. of Seignette solution, and 23 to 24 cc. of diluted wine (approximately 1 per cent), are heated two

minutes to boiling, the precipitated copper allowed to settle, and filtered through a double filter. If the filtrate is greenish, it contains copper; if the filtrate is yellow, it is acidulated, and after cooling a little acetic acid is added and two or three drops potassium ferrocyanid. If a reddish color is formed, copper is still present, and a sufficient amount of sugar has not been added to reduce the 50 cc. of Fehling's solution. The experiment is repeated, adding 1 to 2 cc. more wine if copper was present in the filtrate; if no copper was in the filtrate, 1 cc. less of wine. The experiment is continued till filtrates are obtained containing a difference of 0.1 cc. of wine, one of which contains copper solution and one not. The mean of the two contains the sugar equivalent to 50 cc. of Fehling's solution; 50 cc. of Fehling's solution corresponds to 0.2375 grams of grape sugar.

Polarization.—(1) The wine is decolorized with lead subacetate.

(2) A slight excess of sodium carbonate is added to the filtrate from (1); 2 cc. of a solution of lead subacetate are added to 40 cc. of white wine, and 5 cc. to 40 cc. of red wine, the solution is filtered, and 1 cc. of a saturated solution of sodium carbonate added to 21.0 or 22.5 cc. of the filtrate.

(3) The kind of apparatus used and the length of the tube are to be given, and the results estimated in equivalents of Wild's polaristrobometer, with 200-millimeter tubes, after inversion, are to be considered as containing unfermented glucose (starch sugar) residue.

(5) Rotary power of less than 0.3° to the right shows that impure glucose has not been added.

(6) Wines rotating between 0.3° and 0.5° to the right must be treated by the alcohol method.

(7) Wines rotating strongly to the left must be fermented, and their optical properties then examined.

VIII. *Tannin.*—Determine by the Neubauer and Löwenthal method.

100 cc. of wine is dealcoholized by continued boiling, and the lost weight restored by water. (1) 10 cc. of dealcoholized wine is transferred to a large porcelain evaporating dish, 1 liter of water added, also 10 cc. of dilute H_2SO_4, and then 20 cc. of indigo solution; titrate with $K_2Mn_2O_8$ solution, adding this reagent with great care (drop by drop) towards the end of the reaction. (2) 10 cc. of dealcoholized wine treated with bone black to remove tannin and coloring matter, after standing for a time, is filtered, washed with water, and diluted to 1 liter, and titrated with $K_2Mn_2O_8$ solution, as in (1) (viz, after adding 10 cc. of dilute H_2SO_4 and 20 cc. of indigo).

From the number of cubic centimeters of $K_2Mn_2O_8$ used in (2) subtract the number of cubic centimeters of $K_2Mn_2O_8$ required to oxidize the indigo, and the remainder will give the number of cubic centimeters of $K_2Mn_2O_8$ necessary to oxidize the matters other than tannin and color.

From the number of cubic centimeters of $K_2Mn_2O_8$ used in (1) take the value of the indigo used, and the remainder will be the number of cubic centimeters necessary to oxidize tannin, color, and other oxidizable matter. Subtract from this the number of cubic centimeters of $K_2Mn_2O_8$ from (2) after taking out the value of indigo, and the remainder will be the amount necessary to oxidize the tannin and color. (The amount required for color can be neglected.)

IX. *Potassium bitartrate.*—The determination of potassium bitartrate as such is to be omitted.

X. *Tartaric, malic, and succinic acids.*—(1) According to Schmidt and Hiepe's method.

To 200 cc. of wine evaporated one-half and allowed to cool, basic lead acetate is added to alkaline reaction. The precipitate is filtered off and washed with cold water till the filtrate shows only a slight lead reaction. The precipitate is washed from the filter with a wash bottle into a beaker and treated hot with H_2S till the lead is completely converted into sulphid, filtered hot, and washed with hot water. The filtrate and wash water are evaporated to 50 cc., neutralized with KHO, and again concentrated. An excess of $Ca(C_2H_3O_2)_2$ is added and allowed to stand four to six hours, with frequent stirring. It is then filtered and washed with water till the filtrate and

wash water amount to 100 cc. The calcium tartrate is then converted into caustic lime by strong ignition, and, according to quantity, 10 to 15 cc. of normal HCl added, and the solution diluted and retitrated very accurately. For every cubic centimeter of normal HCl saturated by the caustic lime, 0.075 grams of tartaric acid is to be set down, and to this 0.0286 grams added, representing the total tartaric acid present in 200 cc. wine.

The filtrate from the calcium tartrate is now evaporated to about 20 to 30 cc., allowed to cool, and three times the volume of 96 per cent alcohol added. After standing several hours the precipitate is collected upon a weighed filter, dried at 100° C., and weighed. This represents the lime salts of the malic and succinic acids, and the tartaric acid still remaining in solution, and of the sulphuric acid in the wine. This precipitate is now dissolved in a minimum of HCl, washed with hot water, and then precipitated hot with K_2CO_3, and the $CaCO_3$ filtered off. The filtrate now contains the potash salts of the above-mentioned acids. This is neutralized with acetic acid and evaporated to a small volume, and precipitated hot with $BaCl_2$. This precipitate of succinate and sulphate of barium is treated on the filter with dilute HCl; the filtrate now contains only the succinate of barium, is treated with H_2SO_4 and the Ba precipitated. (The $BaSO_4$ remaining on the filter is ignited and weighed.) The $BaSO_4$ obtained from the succinate solution is filtered off, washed, dried, ignited, and weighed; 223 parts of $BaSO_4$ correspond to 118 parts succinic acid. The succinic and sulphuric acids, as well as the tartaric acid remaining in solution, which was equal to 0.0286 grams, are to be calculated as lime salts and subtracted from the total weight of the lime precipitate. The remainder is calcium malate, of which 172 parts correspond to 134 parts of malic acid.

(2) Determination of tartaric acid according to the modified Berthelot-Fleury method.

Fifty cc. is measured and 10 cc. of same neutralized with caustic potash. The two liquids are now mixed, and one-fifth of the mixture, 10 cc., is taken, and 50 cc. of a mixture of alcohol and ether added and allowed to stand twenty-four hours, and the separated cream of tartar filtered off, dissolved in water, and titrated. The excess of cream of tartar above that obtained in the estimation of tartar alone corresponds to the free tartaric acid.

XI. *Coloring matter.*—(1) Only anilin dyes are to be looked for.

(2) Special attention is to be paid to the spectroscopic behavior of rosanilin dyes as obtained by shaking wines with amylic alcohol before and after saturation with ammonia.

XII. *Inorganic matter* (ash).—Burn in the ordinary manner in a flat platinum dish at as low a heat as possible; repeated moistening, drying, and heating to redness are advisable to get rid of all organic substances.

XIII. *Citric acid.*—Presence to be shown by a qualitative test, as barium citrate.

XIV. *Sulphuric acid.*—To be determined in the wine after adding hydrochloric acid.

XV. *Chlorin.*—To be determined in the nitric acid solution of the burnt residue by Volhard's method.

XVI. *Lime, magnesia, and phosphoric acid.*—These are determined in the ash fused with sodium hydrate and potassium nitrate, the phosphoric acid by the molybdenum method.

XVII. *Potash.*—Either in the wine ash, as the platinum double salt, or in the wine itself, by Kayser's method.

XVIII. *Gums.*—Presence shown by precipitation with alcohol; 4 cc. of wine and 10 cc. of 96 per cent alcohol are mixed. If gum arabic has been added, a lumpy, thick, stringy precipitate is produced; whereas pure wine becomes at first opalescent and then flocculent.

XIX. *Sulphurous acid.*—100 cc. of wine is distilled in a current of carbonic acid after the addition of phosphoric acid. The distillate carefully condensed is oxidized with bromin and the amount of H_2SO_4 determined.

TABLE I.—*Correction to be applied to the reading of Balling's saccharometer for different temperatures.*

	Temp., °F.	Degree Balling.																			
		1°	2°	3°	4°	5°	6°	7°	8°	9°	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°
To be subtracted from the indicated degree.	50	.17	.17	.18	.19	.20	.20	.21	.21	.22	.22	.22	.23	.23	.24	.24	.24	.25	.25	.25	.26	.26
	51	.16	.16	.17	.18	.19	.19	.20	.20	.21	.21	.21	.22	.22	.23	.23	.23	.24	.24	.24	.24	.25
	52	.15	.15	.16	.17	.18	.18	.18	.19	.19	.19	.19	.19	.20	.20	.21	.21	.22	.22	.22	.22	.23
	53	.14	.14	.14	.15	.16	.16	.16	.17	.17	.18	.18	.18	.18	.19	.19	.19	.19	.20	.20	.20	.20
	54	.12	.12	.13	.14	.15	.15	.15	.16	.16	.16	.16	.17	.17	.17	.17	.17	.18	.18	.18	.18	.18
	55	.09	.09	.09	.10	.11	.11	.11	.12	.12	.12	.12	.13	.13	.13	.14	.14	.14	.14	.14	.14	.15
	56	.07	.07	.07	.08	.09	.09	.09	.09	.10	.10	.10	.10	.11	.11	.11	.11	.11	.11	.11	.11	.11
	57	.06	.06	.06	.07	.07	.07	.07	.07	.08	.08	.08	.08	.08	.09	.09	.09	.09	.10	.10	.10	.10
	58	.04	.04	.04	.05	.05	.05	.05	.05	.06	.06	.06	.06	.06	.07	.07	.07	.07	.07	.07	.07	.07
	59	.02	.02	.02	.02	.02	.02	.02	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03
	60																					
To be added to the indicated degree.	61	.02	.02	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03
	62	.06	.06	.07	.07	.08	.08	.08	.08	.08	.08	.09	.09	.09	.09	.09	.09	.09	.09	.09	.09	.09
	63	.09	.10	.10	.11	.11	.11	.11	.11	.11	.11	.12	.12	.12	.12	.12	.12	.12	.12	.13	.12	.12
	64	.12	.12	.13	.13	.14	.14	.14	.14	.15	.15	.15	.15	.16	.16	.17	.17	.17	.17	.17	.17	.17
	65	.14	.15	.15	.16	.17	.17	.17	.17	.18	.18	.18	.19	.19	.19	.20	.20	.20	.20	.20	.20	.20
	66	.17	.17	.18	.19	.20	.20	.21	.21	.21	.22	.22	.23	.23	.23	.24	.24	.24	.24	.24	.24	.24
	67	.20	.21	.21	.22	.23	.23	.23	.24	.24	.25	.25	.25	.26	.26	.27	.27	.27	.27	.27	.27	.27
	68	.22	.23	.24	.25	.26	.26	.27	.28	.28	.29	.29	.30	.30	.31	.31	.31	.31	.31	.31	.31	.31
	69	.25	.26	.27	.28	.29	.29	.30	.31	.31	.32	.32	.33	.33	.34	.34	.34	.34	.34	.34	.34	.34
	70	.28	.29	.30	.31	.32	.32	.33	.34	.34	.35	.35	.36	.36	.37	.37	.37	.37	.38	.38	.38	.38
	71	.31	.32	.33	.34	.35	.35	.36	.37	.37	.38	.38	.39	.39	.40	.40	.40	.40	.40	.40	.40	.41
	72	.34	.35	.36	.37	.38	.39	.40	.41	.42	.42	.42	.43	.43	.43	.44	.44	.44	.44	.44	.44	.44
	73	.37	.38	.40	.41	.42	.43	.44	.44	.45	.46	.46	.46	.46	.47	.47	.47	.47	.47	.47	.47	.47
	74	.40	.42	.43	.45	.46	.47	.48	.48	.49	.50	.50	.50	.51	.51	.51	.51	.51	.52	.52	.52	.52
	75	.44	.46	.47	.49	.50	.51	.52	.52	.53	.54	.54	.54	.55	.55	.56	.56	.56	.57	.57	.58	.58
	76	.47	.48	.50	.52	.53	.54	.55	.55	.56	.57	.57	.58	.58	.59	.60	.60	.61	.61	.61	.62	.62
	77	.50	.52	.54	.56	.57	.58	.59	.60	.60	.61	.61	.62	.62	.63	.63	.63	.64	.64	.64	.65	.65
	78	.53	.54	.56	.58	.60	.61	.62	.62	.63	.64	.64	.65	.65	.66	.66	.66	.67	.67	.68	.68	.68
	79	.57	.58	.60	.62	.64	.65	.66	.66	.67	.68	.68	.69	.69	.70	.70	.70	.71	.71	.71	.72	.72
	80	.60	.62	.64	.66	.68	.69	.70	.70	.71	.72	.72	.72	.73	.73	.74	.74	.75	.75	.75	.76	.76

To be added to the Indicated degree.																						
81	.63	.65	.67	.69	.71	.72	.73	.73	.74	.75	.75	.76	.76	.77	.78	.78	.79	.79	.79	.79	79
82	.67	.69	.71	.73	.76	.76	.77	.77	.78	.78	.79	.80	.80	.81	.82	.82	.83	.83	.84	.84	.84
83	.72	.74	.76	.78	.81	.81	.81	.82	.82	.82	.83	.84	.85	.86	.87	.87	.87	.87	.87	.87	.88
84	.77	.79	.81	.83	.86	.86	.86	.86	.87	.87	.88	.89	.90	.91	.92	.92	.93	.93	.93	.93	.94
85	.83	.85	.87	.89	.91	.91	.92	.92	.93	.93	.94	.95	.96	.97	.98	.98	.98	.99	.99	.99	.99
86	.88	.90	.92	.94	.96	.96	.97	.98	.98	.99	1.00	1.01	1.02	1.02	1.03	1.03	1.04	1.04	1.04	1.05	1.05
87	.94	.96	.98	.99	1.01	1.02	1.03	1.03	1.04	1.05	1.05	1.06	1.07	1.07	1.08	1.08	1.09	1.10	1.10	1.11	1.11
88	.99	1.01	1.03	1.04	1.06	1.07	1.08	1.09	1.10	1.11	1.11	1.12	1.12	1.13	1.13	1.14	1.15	1.15	1.16	1.17	1.17
89	1.05	1.07	1.09	1.10	1.11	1.15	1.13	1.14	1.15	1.16	1.16	1.17	1.17	1.18	1.18	1.19	1.21	1.21	1.22	1.23	1.23
90	1.11	1.13	1.15	1.16	1.17	1.18	1.19	1.20	1.21	1.22	1.22	1.23	1.23	1.24	1.24	1.25	1.27	1.28	1.29	1.30	1.30
91	1.15	1.17	1.19	1.20	1.21	1.22	1.23	1.24	1.26	1.27	1.27	1.27	1.28	1.28	1.29	1.30	1.32	1.33	1.34	1.35	1.36
92	1.20	1.22	1.24	1.25	1.26	1.27	1.28	1.30	1.31	1.32	1.32	1.33	1.33	1.34	1.34	1.35	1.37	1.38	1.39	1.40	1.41
93	1.24	1.26	1.28	1.29	1.31	1.32	1.33	1.34	1.36	1.37	1.37	1.38	1.39	1.39	1.40	1.40	1.42	1.43	1.44	1.44	1.45
94	1.29	1.31	1.33	1.35	1.36	1.37	1.39	1.40	1.41	1.42	1.42	1.43	1.44	1.44	1.45	1.45	1.47	1.47	1.48	1.49	1.50
95	1.33	1.35	1.37	1.39	1.41	1.42	1.44	1.45	1.46	1.47	1.47	1.48	1.49	1.49	1.50	1.50	1.51	1.52	1.53	1.54	1.54
96	1.38	1.40	1.42	1.44	1.46	1.47	1.49	1.50	1.51	1.52	1.52	1.53	1.54	1.54	1.55	1.55	1.56	1.57	1.57	1.58	1.58
97	1.42	1.44	1.46	1.48	1.51	1.52	1.54	1.55	1.56	1.57	1.57	1.58	1.59	1.59	1.60	1.60	1.62	1.62	1.63	1.63	1.64
98	1.47	1.49	1.51	1.54	1.56	1.57	1.58	1.59	1.60	1.62	1.63	1.63	1.64	1.64	1.65	1.65	1.66	1.67	1.67	1.68	1.68
99	1.51	1.53	1.55	1.58	1.61	1.62	1.63	1.64	1.66	1.67	1.68	1.68	1.69	1.70	1.71	1.71	1.72	1.73	1.73	1.73	1.74
100	1.55	1.57	1.59	1.62	1.66	1.67	1.68	1.69	1.72	1.72	1.73	1.73	1.74	1.76	1.77	1.77	1.78	1.79	1.79	1.79	1.80

N. B.—The correction for indications between 21° and 30° Balling and 50° and 70° F. temperature may be made by adding 0.005 to each correction given in the above Table, column 21°, for each increase in degree Balling. For instance, 25° Balling 70° F., correction to be added to column 21° and 70°. 0.02+0.38=0.40.

TABLE II.—*Percentage of alcohol by weight and by volume.*

[Recalculated from Gilpin's, Drinkwater's, Squibb's, etc., determination, by EDGAR RICHARDS.]

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
1.00000	**0.00**	**0.00**	**.99629**	**2.50**	**1.99**	**.99281**	**5.00**	**4.00**	**.98959**	**7.50**	**6.02**
0.99992	.05	.04	622	.55	2.03	274	.05	.04	953	.55	.06
984	.10	.08	615	.60	.07	268	.10	.08	947	.60	.10
976	.15	.12	607	.65	.11	261	.15	.12	940	.65	.14
968	.20	.16	600	.70	.15	255	.20	.16	934	.70	.18
961	.25	.20	593	.75	.19	248	.25	.20	928	.75	.22
953	.30	.24	586	.80	.23	241	.30	.24	922	.80	.26
945	.35	.28	579	.85	.27	235	.35	.28	916	.85	.30
937	.40	.32	571	.90	.31	228	.40	.32	909	.90	.34
930	.45	.36	564	.95	.35	222	.45	.36	903	.95	.38
.99923	**0.50**	**0.40**	**.99557**	**3.00**	**2.39**	**.99215**	**5.50**	**4.40**	**.98897**	**8.00**	**6.42**
915	.55	.44	550	.05	.43	208	.55	.44	891	.05	.46
907	.60	.48	543	.10	.47	202	.60	.48	885	.10	.50
900	.65	.52	336	.15	.51	195	.65	.52	879	.15	.54
892	.70	.56	529	.20	.55	189	.70	.56	873	.20	.58
884	.75	.60	522	.25	.59	182	.75	.60	867	.25	.62
877	.70	.64	515	.30	.64	175	.80	.64	861	.30	.67
869	.85	.67	508	.35	.68	169	.85	.68	855	.35	.71
861	.90	.71	501	.40	.72	162	.90	.72	849	.40	.75
854	.95	.75	494	.45	.76	156	.95	.76	843	.45	.79
.99849	**1.00**	**0.79**	**.99487**	**3.50**	**2.80**	**.99149**	**6.00**	**4.80**	**.98837**	**8.50**	**6.83**
842	.05	.83	480	.55	.84	143	.05	.84	831	.55	.87
834	.10	.87	473	.60	.88	136	.10	.88	825	.60	.91
827	.15	.91	466	.65	.92	130	.15	.92	819	.65	.95
819	.20	.95	459	.70	.96	123	.20	.96	813	.70	.99
812	.25	.99	452	.75	3.00	117	.25	5.00	807	.75	7.03
805	.30	1.03	445	.80	.04	111	.30	.05	801	.80	.07
797	.35	.07	438	.85	.08	104	.35	.09	795	.85	.11
790	.40	.11	431	.90	.12	098	.40	.13	789	.90	.15
782	.45	.15	424	.95	.16	091	.45	.17	783	.95	.19
.99775	**1.50**	**1.19**	**.99417**	**4.00**	**3.20**	**.99085**	**6.50**	**5.21**	**.98777**	**9.00**	**7.23**
768	.55	.23	410	.05	.24	079	.55	.25	771	.05	.27
760	.60	.27	403	.10	.28	072	.60	.29	765	.10	.31
753	.65	.31	397	.15	.32	066	.65	.33	759	.15	.35
745	.70	.35	390	.20	.36	059	.70	.37	754	.20	.39
738	.75	.39	383	.25	.40	053	.75	.41	748	.25	.43
731	.80	.43	376	.30	.44	047	.80	.45	742	.30	.48
723	.85	.47	369	.35	.48	040	.85	.49	736	.35	.52
716	.90	.51	363	.40	.52	034	.90	.53	730	.40	.56
708	.95	.55	356	.45	.56	027	.95	.57	724	.45	.60
.99701	**2.00**	**1.59**	**.99349**	**4.50**	**3.60**	**.99021**	**7.00**	**5.61**	**.98719**	**9.50**	**7.64**
694	.05	.63	342	.55	.64	015	.05	.65	713	.55	.68
687	.10	.67	335	.60	.68	009	.10	.69	707	.60	.72
679	.15	.71	329	.65	.72	002	.15	.73	701	.65	.76
672	.20	.75	322	.70	.76	.98996	.20	.77	695	.70	.80
665	.25	.79	315	.75	.80	990	.25	.81	689	.75	.84
658	.30	.83	308	.80	.84	984	.30	.86	683	.80	.88
651	.35	.87	301	.85	.88	978	.35	.90	678	.85	.92
643	.30	.91	295	.90	.92	971	.40	.94	672	.90	.96
636	.45	.95	288	.95	.96	965	.45	.98	666	.95	8.00

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.98660	10.00	8.04	.98381	12.50	10.08	.98114	15.00	12.13	.97859	17.50	14.19
654	.05	.08	375	.55	.12	108	.05	.17	853	.55	.23
649	.10	.12	370	.60	.16	104	.10	.21	848	.60	.27
643	.15	.16	364	.65	.20	099	.15	.25	843	.65	.31
637	.20	.20	359	.70	.24	093	.20	.29	838	.70	.35
632	.25	.24	353	.75	.28	088	.25	.33	833	.75	.40
626	.30	.29	348	.80	.33	083	.30	.38	828	.80	.44
620	.35	.33	342	.85	.37	078	.35	.42	823	.85	.48
614	.40	.37	337	.90	.41	073	.40	.46	818	.90	.52
609	.45	.41	331	.95	.45	068	.45	.50	813	.95	.56
.98603	10.50	8.45	.98326	13.00	10.49	.98063	15.50	12.54	.97808	18.00	14.60
597	.55	.49	321	.05	.53	057	.55	.58	803	.05	.64
592	.60	.53	315	.10	.57	052	.60	.62	798	.10	.68
586	.65	.57	310	.15	.61	047	.65	.66	793	.15	.73
580	.70	.61	305	.20	.65	042	.70	.70	788	.20	.77
575	.75	.65	299	.25	.69	037	.75	.75	783	.25	.81
569	.80	.70	294	.30	.74	032	.80	.79	778	.30	.85
563	.85	.74	289	.35	.78	026	.85	.83	773	.35	.89
557	.90	.78	283	.40	.82	021	.90	.87	768	.40	.94
552	.95	.82	278	.45	.86	016	.95	.91	763	.45	.98
98546	11.00	8.86	.98273	13.50	10.90	.98011	16.00	12.95	.97758	18.50	15.02
540	.05	.90	267	.55	.94	005	.05	.99	753	.55	.06
535	.10	.94	262	.60	.98	001	.10	13.03	748	.60	.10
529	.15	.98	256	.65	11.02	.97996	.15	.08	743	.65	.14
524	.20	9.02	251	.70	.06	991	.20	.12	738	.70	.18
518	.25	.07	246	.75	.11	986	.25	.16	733	.75	.22
513	.30	.11	240	.80	.15	980	.30	.20	728	.80	.27
507	.35	.15	235	.85	.19	975	.35	.24	723	.85	.31
502	.40	.19	230	.90	.23	970	.40	.29	718	.90	.38
496	.45	.23	224	.95	.27	965	.45	.33	713	.95	.39
.98491	11.50	9.27	98.219	14.00	11.31	.97960	16.50	13.37	.97708	19.00	15.43
485	.55	.31	214	.05	.35	955	.55	.41	703	.05	.47
479	.60	.35	209	.10	.39	950	.60	.45	698	.10	.51
474	.65	.39	203	.15	.43	945	.65	.49	693	.15	.55
468	.70	.43	198	.20	.47	940	.70	.53	688	.20	.59
463	.75	.47	193	.25	.52	935	.75	.57	683	.25	.63
457	.80	.51	188	.30	.56	929	.80	.62	678	.30	.68
452	.85	.55	182	.35	.60	924	.85	.66	673	.35	.72
446	.90	.59	177	.40	.64	919	.90	.70	668	.40	.76
441	.95	.63	172	.45	.68	914	.95	.74	663	.45	80
.98435	12.00	9.67	.98167	14.50	11.72	.97909	17.00	13.78	.97658	19.50	15.84
430	.05	.71	161	.55	.76	904	.05	.82	653	.55	.88
424	.10	.75	156	.60	.80	899	.10	.86	648	.60	.93
419	.15	.79	151	.65	.84	894	.15	.90	643	.65	.97
413	.20	.83	146	.70	.88	889	.20	.94	638	.70	16.01
408	.25	.87	140	.75	.93	884	.25	.98	633	.75	.05
402	.30	.92	135	.80	.97	879	.30	14.03	628	.80	.09
397	.35	.96	130	.85	12.05	874	.35	.07	623	.85	.14
391	.40	10.00	125	.90	.05	869	.40	.11	618	.90	.18
386	.45	.04	119	.95	.09	864	.45	.15	613	.95	.22

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.97608	20.00	16.26	.97355	22.50	18.34	.97097	25.00	20.43	.96828	27.50	22.54
603	.05	.30	350	.55	.38	092	.05	.47	822	.55	.58
598	.10	.34	345	.60	.42	086	.10	.51	816	.60	.62
593	.15	.38	340	.65	.47	081	.15	.56	811	.65	.67
588	.20	.42	335	.70	.51	076	.20	.60	805	.70	.71
583	.25	.46	330	.75	.55	071	.25	.64	800	.75	.75
578	.30	.51	324	.80	.59	065	.30	.68	794	.80	.79
573	35	.58	319	.85	.63	060	.35	.72	789	.85	.83
568	.40	.59	314	.90	.68	055	.40	.77	783	.90	.88
563	45	.63	309	.95	.72	049	.45	.81	778	.95	.92
.97558	20.50	16.67	.97304	23.00	18.76	.97044	25.50	20.85	.96772	28.00	22.96
552	.55	.71	299	.05	.80	039	.55	.89	766	.05	23.00
547	.60	.75	294	.10	.84	033	.60	.93	761	.10	.04
542	.65	.80	289	.15	.88	028	.65	.98	755	.15	.09
537	.70	.84	283	.20	.92	023	.70	21.02	749	.20	.13
532	.75	.88	278	.25	.96	018	.75	.06	744	.25	.17
527	.80	.92	273	.30	19.01	012	.80	.10	738	.30	.21
522	.85	.96	268	.35	.05	007	.85	.14	732	.35	.25
517	.90	17.01	263	.40	.09	001	.90	.19	726	.40	.30
512	.95	.05	258	.45	.13	.96996	.95	.23	721	.45	.34
.97507	21.00	17.09	.97253	23.50	19.17	.96991	26.00	21.27	.96715	28.50	23.38
502	.05	.13	247	.55	.21	986	.05	.31	709	.55	.42
497	.10	.17	242	.60	.25	980	.10	.35	704	.60	.47
492	.15	.22	237	.65	.30	975	.15	.40	698	.65	.51
487	.20	.26	232	.70	.34	969	.20	.44	692	.70	.55
482	.25	.30	227	.75	.38	964	.25	.48	687	.75	.60
477	.30	.34	222	.80	.42	959	.30	.52	681	.80	.64
472	.35	.38	216	.85	.46	953	.35	.56	675	.85	.68
467	.40	.43	211	.90	.51	949	.40	.61	669	.90	.72
562	.45	.47	206	.95	.55	942	.45	.65	664	.95	.77
.97457	21.50	17.51	.97201	24.00	19.59	.96937	26.50	21.69	.96658	29.00	23.81
451	.55	.55	196	.05	.63	932	.55	.73	652	.05	.85
446	.60	.59	191	.10	.67	926	.60	.77	646	.10	.89
441	.65	.63	185	.15	.72	921	.65	.82	640	.15	.94
436	.70	.67	180	.20	.76	915	.70	.86	635	.20	.98
431	.75	.71	175	.25	.80	910	.75	.90	629	.25	24.02
426	.80	.76	170	.30	.84	905	.80	.94	623	.30	.06
421	.85	.80	165	.35	.88	899	.85	.98	617	.35	.10
416	.90	.84	159	.40	.93	894	.90	22.03	611	.40	.15
411	.95	.88	154	.45	.97	888	.95	.07	605	.45	.19
.97406	22.00	17.92	.97149	24.50	20.01	.96883	27.00	22.11	.96600	29.50	24.23
401	.05	.96	144	.55	.05	877	.05	.15	594	.55	.27
396	.10	18.00	139	.60	.09	872	.10	.20	587	.60	.32
391	.15	.05	133	.65	.14	866	.15	.24	582	.65	.36
386	.20	.03	128	.70	.18	861	.20	.28	576	.70	.40
381	.25	.13	123	.75	.22	855	.25	.33	570	.75	.45
375	.30	.17	118	.80	.26	850	.30	.37	564	.80	.49
370	.35	.21	113	.85	.30	844	.35	.41	559	.85	.53
365	.40	.26	107	.90	.35	839	.40	.45	553	.90	.57
360	.45	30	102	.95	.39	833	.45	.50	547	.95	.62

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.96541	**30.00**	**24.66**	**.96235**	**32.50**	**26.80**	**.95910**	**35.00**	**28.96**	**.95560**	**37.50**	**31.14**
535	.05	.70	229	.55	.84	903	.05	29.00	552	.55	.18
529	.10	.74	222	.60	.89	896	.10	.05	545	.60	.23
523	.15	.79	216	.65	.93	889	.15	.09	538	.65	.27
517	.20	.83	210	.70	.97	883	.20	.13	531	.70	.32
511	.25	.87	204	.75	27.02	876	.25	.18	523	.75	.36
505	.30	.91	197	.80	.06	869	.30	.22	516	.80	.40
499	.35	.95	191	.85	.10	862	.35	.26	509	.85	.45
493	.40	25.00	185	.90	.14	855	.40	.30	502	.90	.49
487	.45	.04	178	.95	.19	848	.45	.35	494	.95	.54
.96481	**30.50**	**25.08**	**.96172**	**33.00**	**27.23**	**.95842**	**35.50**	**29.39**	**.95487**	**38.00**	**31.58**
475	.55	.12	166	.05	.27	835	.55	.43	480	.05	.63
469	.60	.17	159	.10	.32	828	.60	.48	472	.10	.67
463	.65	.21	153	.15	.36	821	.65	.52	465	.15	.72
457	.70	.25	146	.20	.40	814	.70	.57	457	.20	.76
451	.75	.30	140	.25	.45	807	.75	.61	450	.25	.81
445	.80	.34	133	.30	.49	800	.80	.65	442	.30	.85
439	.85	.38	127	.35	.53	794	85	.70	435	.35	.90
433	.90	.42	120	.40	.57	787	.90	.74	427	.40	.94
427	.95	.47	114	.45	.62	780	.95	.79	420	.45	.99
.96421	**31.00**	**25.51**	**.96108**	**33.50**	**27.66**	**.95773**	**36.00**	**29.83**	**.95413**	**8.50**	**32.03**
415	.05	.55	101	.55	.70	766	.05	.87	405	.55	.07
409	.10	.60	095	.60	.75	759	.10	.92	398	.60	.12
403	.15	.64	088	.65	.79	752	.15	.96	390	.65	.16
396	.20	.68	082	.70	.83	745	.20	30.00	383	.70	.20
390	.25	.73	075	.75	.88	738	.25	.05	375	.75	.25
384	.30	.77	069	.80	.92	731	.30	.09	368	.80	.29
378	.35	.81	062	.85	.96	724	.35	.13	360	.85	.33
372	.40	.85	056	.90	28.00	717	.40	.17	353	.90	.37
366	.45	.90	049	.95	.05	710	.45	.22	345	.95	.42
.96360	**31.50**	**25.94**	**.96043**	**34.00**	**28.09**	**.95703**	**36.50**	**30.26**	**.95338**	**39.00**	**32.46**
353	.55	.98	036	.05	.13	695	.55	.30	330	.05	.50
347	.60	26.03	030	.10	.18	688	.60	.35	323	.10	.55
341	.65	.07	023	.15	.22	681	.65	.39	315	.15	.59
335	.70	.11	016	.20	.26	674	.70	.44	307	.20	.64
329	.75	.16	010	.25	.31	667	.75	.48	300	.25	.68
323	.80	.20	003	.30	.35	660	.80	.52	292	.30	.72
316	.85	.24	.95996	.35	.39	653	.85	.57	284	.35	.77
310	.90	.28	990	.40	.43	646	.90	.61	277	.40	.81
304	.95	.33	983	.45	.48	639	.95	.66	269	.45	.86
.96298	**32.00**	**26.37**	**.95977**	**34.50**	**28.52**	**.95632**	**37.00**	**30.70**	**.95262**	**39.50**	**32.90**
292	.05	.41	970	.55	.56	625	.05	.74	254	.55	.95
285	.10	.46	963	.60	.61	618	.10	.79	246	.60	.99
279	.15	.50	957	.65	.65	610	.15	.83	239	.65	33.04
273	.20	.54	950	.70	.70	603	.20	.88	231	.70	.08
267	.25	.59	943	.75	.74	596	.25	.92	223	.75	.12
260	.30	.63	937	.80	.78	589	.30	.96	216	.80	.17
254	.35	.67	930	.85	.83	581	.35	31.01	208	.85	.22
248	.40	.71	923	.90	.87	574	.40	.05	200	.90	.27
241	.45	.76	917	.95	.92	567	.45	.10	193	.95	.31

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.95185	40.00	33.35	.94786	42.50	35.58	.94364	45.00	37.84	.93916	47.50	40.13
177	.05	.39	778	.55	.63	355	.05	.89	906	.55	.18
169	.10	.44	770	.60	.67	346	.10	.93	898	.60	.22
161	.15	.48	761	.65	.72	338	.15	.98	888	.65	.27
154	.20	.53	753	.70	.76	329	.20	38.02	879	.70	.32
146	.25	.57	745	.75	.81	320	.25	.07	870	.75	.37
138	.30	.61	737	.80	.85	311	.30	.12	861	.80	.41
130	.35	.66	729	.85	.90	302	.35	.16	852	.85	.46
122	.40	.70	720	.90	.94	294	.40	.21	842	.90	.51
114	.45	.75	712	.95	.99	285	.45	.25	833	.95	.55
.95107	40.50	33.79	.94704	43.00	36.03	.94276	45.50	38.30	.93824	48.00	40.60
099	.55	.84	696	.05	.08	267	.55	.35	815	.05	.65
091	.60	.88	687	.10	.12	258	.60	.39	805	.10	.69
083	.65	.93	679	.15	.17	250	.65	.44	796	.15	.74
075	.70	.97	670	.20	.21	241	.70	.48	786	.20	.78
067	.75	34.02	662	.25	.26	232	.75	.53	777	.25	.83
059	.80	.06	654	.30	.30	223	.80	.57	768	.30	.88
052	.85	.11	645	.35	.35	214	.85	.62	758	.35	.92
044	.90	.15	637	.40	.39	206	.90	.66	749	.40	.97
036	.95	.20	628	.45	.44	197	.95	.71	739	.45	41.01
.95028	41.00	34.24	.94620	43.50	36.48	.94188	46.00	38.75	.93730	48.50	41.06
020	.05	.28	612	.55	.53	179	.05	.80	721	.55	.11
012	.10	.33	603	.60	.57	170	.10	.84	711	.60	.15
004	.15	.37	595	.65	.62	161	.15	.89	702	.65	.20
.94996	.20	.42	586	.70	.66	152	.20	.93	692	.70	.24
988	.25	.46	578	.75	.71	143	.25	.98	683	.75	.29
980	.30	.50	570	.80	.75	134	.30	39.03	679	.80	.34
972	.35	.55	561	.85	.80	125	.35	.07	664	.85	.38
964	.40	.59	553	.90	.84	116	.40	.12	655	.90	.43
956	.45	.64	544	.95	.89	107	.45	.16	645	.95	.47
.94948	41.50	34.68	.94536	44.00	36.93	.94098	46.50	39.21	.93636	49.00	41.52
940	.55	.73	527	.05	.98	089	.55	.26	626	.05	.57
932	.60	.77	519	.10	37.02	080	.60	.30	617	.10	.61
924	.65	.82	510	.15	.07	071	.65	.35	607	.15	.66
916	.70	.86	502	.20	.11	062	.70	.39	598	.20	.71
908	.75	.91	493	.25	.16	053	.75	.44	588	.25	.76
900	.80	.95	484	.30	.21	044	.80	.49	578	.30	.80
892	.85	35.00	476	.35	.25	035	.85	.53	569	.35	.85
884	.90	.04	467	.40	.30	026	.90	.58	559	.40	.90
876	.95	.09	459	.45	.34	017	.95	.62	550	.45	.94
.94868	42.00	35.13	.94450	44.50	37.39	.94008	47.00	39.67	.93540	49.50	41.99
860	.05	.18	441	.55	.44	.93999	.05	.72	530	.55	42.04
852	.10	.22	433	.60	.48	990	.10	.76	521	.60	.08
843	.15	.27	424	.65	.53	980	.15	.81	511	.65	.13
835	.20	.31	416	.70	.57	671	.20	.85	502	.70	.18
827	.25	.36	407	.75	.62	962	.25	.90	492	.75	.23
819	.30	.40	398	.80	.66	953	.30	.95	482	.80	.27
811	.35	.45	390	.85	.71	944	.35	.99	473	.85	.32
802	.40	.49	381	.90	.76	934	.40	40.04	463	.90	.37
794	.45	.54	373	.95	.80	925	.45	.08	454	.95	.41

TABLE OF ATOMIC WEIGHTS.

[Issued December 6, 1890.]

Revised by F. W. CLARKE, *Chief Chemist of the United States Geological Survey.*

[This table represents the latest and most trustworthy results, reduced to a uniform basis of comparison, with oxygen=16 as starting point of the system. No decimal places representing large uncertainties are used. When values vary, with equal probability on both sides, so far as our present knowledge goes, as in the case of cadmium (111.8 and 112.2), the mean value is given in the table.]

Name.	Symbol.	Atomic weight.	Name.	Symbol.	Atomic weight.
Aluminum	Al	27	Molybdenum	Mo	96
Antimony	Sb	120	Nickel	Ni	58.7
Arsenic	As	75	Nitrogen	N	14.03
Barium	Ba	137	Osmium	Os	190.8
Bismuth	Bi	208.9	Oxygen[4]	O	16
Boron	B	11	Palladium	Pd	106.6
Bromin	Br	79.95	Phosphorus	P	31
Cadmium	Cd	112	Platinum	Pt	195
Cesium	Cs	132.9	Potassium	K	39.11
Calcium	Ca	40	Rhodium	Rh	103
Carbon	C	12	Rubidium	Rb	85.5
Cerium	Ce	140.2	Ruthenium	Ru	101.6
Chlorin	Cl	35.45	Samarium	Sm	150
Chromium	Cr	52.1	Scandium	Sc	44
Cobalt	Co	59	Selenium	Se	79
Columbium[1]	Cb	94	Silicon	Si	28.4
Copper	Cu	63.6	Silver	Ag	107.92
Didymium[2]	Di	142.3	Sodium	Na	23.05
Erbium	Er	166.3	Strontium	Sr	87.6
Fluorin	F	19	Sulphur	S	32.06
Gallium	Ga	69	Tantalum	Ta	182.6
Germanium	Ge	72.3	Tellurium	Te	125
Glucinum[3]	Gl	9	Terbium	Tb	159.5
Gold	Au	197.3	Thallium	Tl	204.18
Hydrogen	H	1.007	Thorium	Th	232.6
Indium	In	113.7	Tin	Sn	119
Iodin	I	126.85	Titanium	Ti	48
Iridium	Ir	193.1	Tungsten	W	184
Iron	Fe	56	Uranium	U	239.6
Lanthanum	La	138.2	Vanadium	V	51.4
Lead	Pb	206.95	Ytterbium	Yb	173
Lithium	Li	7.02	Yttrium	Yt	89.1
Magnesium	Mg	24.3	Zinc	Zn	65.3
Manganese	Mn	55	Zirconium	Zr	90.6
Mercury	Hg	200			

[1] Has priority over Niobium.
[2] Now split into Neo- and Praseo-Didymium.
[3] Has priority over Beryllium.
[4] Standard or basis of the system.

INDEX.

A.

Page.

Absolute method for nitrogen ---- 198
Abstracts, committee on ---- 192
motion to appoint ---- 104
discussion in regard to making ---- 102
Acid digestion of soil, provisional method ---- 227
in fermented liquors, official method ---- 233
sugar solutions ---- 149
wine ---- 174, 175, 178
methods of standardizing ---- 75
discussion of ---- 90
soluble material in soils, provisional method ---- 227
standard, comparison of ---- 74
against tetra-oxalate ---- 77
official method of preparing ---- 200
standardization by acid potassium tartrate ---- 84
potassium tetra-oxalate ---- 76, 82
summary of results of methods ---- 77
paper by E. M. Parsons ---- 80
use of indicators ---- 77, 78
volatile, official methods for ---- 219, 221, 233
Address of Hon. Edwin Willits ---- 98
president, Mr. N. T. Lupton ---- 2
Adriance, D., remarks on work on association samples ---- 131
Albuminoid nitrogen, official method ---- 215
Alcohol ---- 172, 174–180
official method ---- 232
table of specific gravities ---- 238
Alkalis in ash, provisional method ---- 232
sugar products ---- 168
soils, provisional method ---- 228
Allihn's dextrose method ---- 210
table for dextrose ---- 210
Alumina cream ---- 213
in soils, provisional method ---- 227
Ammonium, citrate solution ---- 196
molybdate solution ---- 196
nitrate solution ---- 197
Anderson, J. T., experiments on ash determinations in cattle foods ---- 134
moisture determinations in cattle foods 132
report on cattle foods ---- 127
Anhalt mines, potash method used at ---- 63

Page.
Anilin colors in wine ---- 235
Areo-picnometer ---- 168
Ash. (*See also* Soils and Ash.)
analysis, provisional methods ---- 230
ferric oxid method ---- 164
in cattle foods ---- 115, 118, 130, 134
cheese, provisional method ---- 225
fermented liquors, official method ---- 235
foods and feeding stuffs, official method ---- 214
milk, official method ---- 224
sugar products ---- 140, 143, 161
official methods ---- 205, 206
Atomic weights, table of ---- 243

B.

Babcock's method for fat in cheese, tests ---- 18, 19
Babcock, S. M., report on dairy products ---- 7
work on cheese ---- 16
Leffman-Beam method ---- 9
Bartlett, S. C., work on Leffman-Beam method ---- 9
Benzoic acid method for ash ---- 140, 161
official ---- 206
Blalock, T. L., work on potash samples ---- 60
Boneblack error ---- 154
Brackett, R. N., work on phosphoric acid samples ---- 26
Brix spindle, table of correction for temperature ---- 236
Butter analysis, association work on ---- 11
comparison of official and Leffman-Beam methods for ---- 8
Leffman-Beam method for ---- 7
official methods for ---- 216

C.

Capillarity of soils, provisional method ---- 226
Carbohydrates, tests for certain ---- 168
Carbon in ash, provisional method ---- 231
Carbonic acid in ash, provisional methods ---- 231
soil, provisional method ---- 230
Carpenter, F. B., work done on phosphoric acid ---- 26
Carr, O., remarks on analysis of wine samples ---- 180
Casein, factor for ---- 22
in butter, official method ---- 217
cheese, provisional method ---- 225
Cathcart, C. S., remarks on work on nitrogen samples ---- 71
Cattle foods. (*See also* Foods and Feeding Stuffs.)
analyses of association samples (Anderson) ---- 128
(Jordan) ---- 115, 118, 123
circular regarding samples sent out (Jordan) ---- 114
discussion of reports ---- 136
preparation of samples (Anderson) ---- 127
recommendations of reporter (Anderson) ---- 135
reporter's comments on work on association samples (Anderson) ---- 132

Page.
Cattle foods, reporter's comments on work on association samples (Jordan) 117, 120, 126
report of J. T. Anderson 127
W. M. Jordan 114
Cheese analysis, association work on 12, 19
provisional methods for 224
determination of fat by different methods 18
Short's method 15
factor for calculating protein 21, 23
sampling, difficulties of 12
work on drying of 13, 14
Chemists (official) connected with agricultural colleges 188
the State agricultural experiment stations 186
U. S. Department of Agriculture 190
Treasury Department 191
resolution relative to list of 101
Chlorin in ash, provisional method 327
fermented liquors, official method 235
soil, provisional method 231
Citrate soluble phosphoric acid, official method 196
solution, official method of making 196
Citric acid in wine 235
Clerget's method 144, 145, 154
official 213
Cocoa, estimation of sugar in 161
Colby, G. F., remarks on analysis of wine samples 179
Collingwood, C. B., remarks on analysis of wine samples 174
Coloring matter in wine 235
Committee on abstracts 102, 104, 192
international chemical congress 100, 192
nomination of officers 93
report 100
Committees for 1892–'93 192
Condensers, comparison of glass and tin 10, 11
discussion on 20
Constitution of association 193
Copper, electrolytic methods for 143, 170, 208, 212
official 208
Cottonseed meal, occurrence of pyro and metaphosphoric acids in 50
Crampton, C. A., report on fermented liquors 171
Crude fiber, comparative results by different methods 122
Hönig method 121
in cattle foods 120, 131, 135
official method 215
Curtis, H. E., work on phosphoric acid 25

D.

Dairy products, official methods for 216
report on 7
discussion of 20
Davidson, R. J., work on association phosphoric acid samples 29
De Roode, on use of permanganate in Kjeldahl method 85

Page.
De Roode, remarks on indicators ---- 78
work on phosphoric acid samples ---- 84
Dextrose, determination of ---- 137, 210
Drying cattle foods, experiments on ---- 116
in vacuo ---- 169

E.

Eaton, E. N., remarks on work on association samples ---- 96, 177
Editing of report, remarks on ---- 101
Election of officers ---- 100
Electrolytic copper determinations ---- 143, 170, 208, 212
Ether extract. (*See also* Fat.)
experiments on Patterson's method ---- 123, 124
in cattle foods ---- 115, 123, 130
official methods ---- 214, 216, 223, 225
saponification number ---- 125
Ewell, E. E., on electrolytic estimation of copper ---- 170
remarks on analysis of association sugar samples ---- 144
Extract, in fermented liquors, official method ---- 233
wines ---- 172, 174–180

F.

Failyer, G. H., remarks on analysis of association wine samples ---- 174
Falkenbach, F. J., work on association phosphoric acid samples ---- 30, 33, 35
Farrington, E. H., work on association samples ---- 28, 35, 116, 118, 123, 130
Fat. (*See also* Ether Extract.)
in butter, official method ---- 216
cheese, provisional method ---- 225
milk, official methods ---- 223
Fatty acids, melting before distillation ---- 12
Fehling solution ---- 206
Fermented liquors, circular regarding association samples ---- 172
miscellaneous work on ---- 180
official methods ---- 232
recommendations of reporter ---- 183
reporter's comments on results on association samples ---- 173
report on ---- 171
discussion of ---- 184
results obtained on association samples ---- 173
Fiber, separation from starch ---- 115
Fields, J. W., remarks on indicators ---- 78
work on association samples ---- 129–131
Film drying ---- 182
Food and feeding stuffs, official methods ---- 214
Formanek, electrolytic method for copper ---- 143, 212
Fries, A. W., work on association phosphoric acid samples ---- 24

G.

Glycerol in fermented liquors, official method ---- 233
Glycerol-soda method for crude fiber ---- 121
of saponification ---- 7

Page.
Gums in fermented liquors, official method 235
Gunning and Kjeldahl methods, comparative results by 70
method 68, 85
adaptation to nitrates 86
official 202
paper by E. B. Voorhees 85

H.

Hardin, M. B., paper on occurrence of meta and pyrophosphoric acids in cottonseed meal 50
Harrington, H. H., report on soils and ash 93
Hartwell, B. L., and H. J. Wheeler, paper on use of ammonium nitrate 53
Heberly, G. J. A., work on association phosphoric acid samples 30
Heilmann, W. H., work on Leffman-Beam method 9
Herzfeld's table for invert sugar 208
Hills, J. L., work on phosphoric acid 29
Hönig method for crude fiber 121
Hopkins, C. G., work on association cattle-food samples 130, 131
Huston, H. A., paper on water determination in association phosphate samples 47
remarks on analysis of association wine samples 176

I.

Indicators, official 200
use of 77, 78
Insoluble phosphoric acid, official method 195
Inversion of cane sugar 137, 144, 145, 151
Invert sugar, crystalline magma of 155
polarization of 151, 152
Iodin absorption number, official method 221
Iron in ash, provisional method 232
soils, provisional method 227

J.

Jenter, C. G., remarks on indicators 78
Jordan, W. M., report on cattle foods 114

K.

Kedzie, R. C., work on association samples 27, 130, 175
Kilgore, B. W., remarks on analysis of association wine samples 175
Kjeldahl method, comparison with Gunning method 68, 70
modified for nitrates, official 202
official 200
Knisely, A. L., method of standardizing acid 75
Koettstorfer's method applied to ether extract 125
Krug, W. H., work on association samples 129, 130

L.

Lead, error in sugar analysis 146, 160
subacetate, preparation of 213
Leffman and Beam method 7
discussion of 20

Page.
Letter of German Kali syndicate, quotation from ... 61
transmittal ... 111
Levulose ... 137
Lime in ash, provisional method ... 232
soils, provisional method ... 228
wine, official method ... 235
Lindet-Clerget method ... 214
Lindo-Gladding method for potash ... 197
Liquors. (*See* Fermented Liquors.)
List of members and others in attendance ... 1
official chemists ... 186, 189, 190
Lord, N. W., determination of water in association phosphate samples ... 31
report on phosphoric acid ... 23
Lupton, N. T., address of ... 2
Lyon, T. L., work on association samples ... 15, 144

M.

McDonald, H. B., work on association potash samples ... 60
McElroy, K. P., work on association samples ... 8, 13, 24
Magnesia in ash, provisional method ... 232
fermented liquors, official method ... 235
soils, provisional method ... 228
mixture ... 197
Magnesium nitrate solution ... 197
Malic acid in wine, official method ... 234
Manganese in ash, provisional method ... 232
soils, provisional method ... 228
Meeting of 1893, discussion in regard to location of ... 104
Meissl and Hiller's table for invert sugar ... 210
Melting point, butter, official methods ... 218
Meng, J. S., work done on phosphoric acid ... 26, 27
Methods of analysis adopted as official ... 195
Microscopic examination of butter ... 216
Milk, official methods for ... 223
Miscellaneous business ... 100
Miscellaneous determinations in wine ... 180
Moisture. (*See also* Water.)
in ash, provisional method ... 231
cattle foods ... 114, 115, 129, 132
foods and feeding stuffs ... 214
soils, provisional method ... 226
Molybdate solution ... 196
Moore, R. B., work on association phosphoric acid samples ... 30
Morse, F. W., work on association phosphoric acid samples ... 28

N.

Nitrogen absolute method, official ... 198
comparative results by Kjeldahl and Gunning methods ... 70, 87
comparison of standard acids ... 74
discussion of methods ... 79, 89
Gunning method ... 85, 202
adapted to nitrates ... 86

Page.
Nitrogen in cattle foods 124, 131
milk, official method 223
nitrates, Ulsch method 88
soil, provisional method 229
Kjeldahl method 200
modification of Scovell-Jodlbaur method 86
official methods for 198
recommendations of reporter 78
report on 67
results by T. C. Trescot 79
Ulsch method 73
Ruffle method 202
soda-lime method 203
summary of results on association samples 70, 72, 73, 74
tables of results on association samples 71, 72, 73
Ulsch method for nitrates 88
use of indicators 77
permanganate in Gunning method 72
Kjeldahl method 71, 85
potassium sulphate 89
Noble, R. E., work on association phosphoric acid samples 26

O.

O'Brine, David, work on Leffman-Beam method 8
Officers, committee on nomination of 93
report of 100
election of 100
list for 1892-'93 192
Official methods 195
for butter 216
dairy products 216
foods and feeding stuffs 214
milk 223
nitrogen 198
phosphoric acid 195
potash 197
sampling 195
sugar analysis 204
Order of business, report of committee on 2

P.

Parsons, E. M., paper on standardization of acids 80
remarks on indicators 78
Patrick, G. E., work on Leffman-Beam method 9
Patterson, H. J., ether extract in cattle food samples 131
Patterson, L. G., work on phosphoric acid 28
Patterson's method for ether extract 123
Payne, G. F., report on potash 58
Pellet's continuous polarization tube 138, 167
Penny, C. L., work on association samples 8, 178
Perkins, W. R., work on association phosphoric acid samples 28

Page.
Peter, A. M., remarks on official soil and ash work 94
Phosphates of Florida, analyses of 6
Phosphoric acid .....
comments by reporter on work on association samples 31, 34, 37
description of association samples 24
discussion on results of work on 54
water determinations 54
in ash, provisional method 231
fermented liquors, official method 235
soils, provisional methods 227, 228
insoluble, discussion of methods for 57
results on, by reporter 37
use of citrate in wash water 37, 58
official methods for 195
preparation of reagents 196
results on 39
pyro and meta, occurrence in cottonseed meal 50
report on 23
samples .....
mechanical analysis of 36
total, comparative results by different methods 45
water determination in association samples, paper by H. A. Huston 47
Picnometer 168
Potash, criticism of official method by Dr. Zimmermann 64
description of various methods of analysis 63
factory method of mixing fertilizers containing 62
in ash, provisional method 232
fermented liquors, official method 235
soils, provisional method 228
Lindo-Gladding method for 197
official factors for 198
methods for determination of 197
oxalic acid method 63
precipitation method 63
preparation of association samples 58
recommendations of reporter on 61
report 58
discussion of 65
table of results on association samples 59
Preparation of ash samples 23
President, annual address of 2
Programme 2
Protein in foods and feeding stuffs 215
Provisional methods for ash analysis 230

R.

Raffinose, determination of 157, 158
Reducing sugars 138, 143, 158, 159, 180
official methods 206
Reporters, list of those appointed for 1892–'93 192
Robinson, Norman, work on association potash samples 60
Ross, B. B., report on sugar 137

Page.
Rotary power of dextrose 152
invert sugar 152
levulose 152
sugar solution 151
Ruffle method, official 202

S.

Salt in butter, official method 217
Samples, official method for preparation of 195
Sampling butter 216
cheese, provisional method 224
soil, provisional method 225
sugar juices 168
Sand and silica in ash, provisional method 231
Scovell-Jodlbaur method, modification of 86
Scovell, M. A., use of potassium sulphate in place of mercuric oxid 89
Shepard, J. H., work on association cattle food sample 130, 131
Shiver, F. S., work on association samples 26, 60, 178
Short's method for fat in cheese 15, 225
Silica in ash, provisional method 231
Snyder, Harry, work on cheese 16
Soda in ash, provisional method 232
soils, provisional method 228
Soda-lime method, official 203
Sodium chlorid, use in potash method 59
Soils and ash, discussion of methods 105
reporter's comments on results on association samples 96
provisional methods 225
recommendations of reporter 97
report on 93
sampling 106, 108
sifting of samples 97, 106
table of analyses of association samples 95
Soxhlet's method for reducing sugar 207
Specific gravity of association wine samples ..... 174 to 180
of butter, official method 217
official method 232
Stone, E. P., work on phosphoric acid 28
Street, J. P., Ulsch method for nitrogen in nitrates 88
Stutzer's method for albuminoid nitrogen 215
Succinic acid in wine, official method 234
Sugar, comments by reporter on analyses of association samples 142, 144
detection of small quantities of 155
German method for sucrose 213
in beets 139, 150
fermented liquors, official method 233
milk, official method 223
influence of boiling solutions 156
inversion of 137, 138, 145, 146
literature of 137, 150
official methods for analysis 204
oxidation of 151

Page.
Sugar, raw, organic constituents of ---- 165
recommendations of reporter on ---- 149
report on ---- 137
discussion of ---- 168
rotation of ---- 137, 145
solubility of ---- 155
sucrose ---- 212
table of analyses of association samples ---- 141
Sulphuric acid in ash, provisional method ---- 232
soils, provisional method ---- 227, 228
Sulphurous acid in fermented liquors, official method ---- 235
Sweetser, W. S., remarks on work on potash samples ---- 60

T.

Tannin in wine, official method ---- 234
Tartaric acid in wine, official method ---- 234
Teller, G. L., method of standardizing acid ---- 75, 76
remarks on indicators ---- 78
work on association wine samples ---- 179
Tetra-oxalate, character of commercial samples ---- 83
preparation of pure ---- 83
potassium ---- 82
hygroscopic qualities of ---- 84
Tin condensers, comparison with glass ---- 11
discussion of ---- 21
Total solids in sugar products ---- 205
wine ---- 181
Trescot, T. C., nitrogen results by ---- 79

U.

Ulsch method for nitrogen in nitrates ---- 88
results obtained by ---- 73

V.

Van Slyke, L. L., report on nitrogen ---- 67
Viets, W. J., work on phosphoric acid samples ---- 32, 35
Volatile acids, butter, official methods ---- 219
in fermented liquors, official method ---- 233
matter in soils, provisional method ---- 226
Voorhees, E. B., Gunning-Kjeldahl method ---- 85
work on association nitrogen samples ---- 71
Voorhees, L. A., ether extract of association cattle-food samples ---- 130
Vote of thanks ---- 105

W.

Waller, Elwyn, work on association potash samples ---- 60
Water. (*See also* Moisture.)
in butter, official method ---- 216
cheese, provisional method ---- 224
milk, official method ---- 223
sugar products ---- 139, 142, 164, 204

Page.
Water, retentive capacity of soils for, provisional method 226
soluble matter in soils, provisional method 226
phosphoric acid, official method 195
Watson, T. L., work on association phosphoric acid samples 29
Wheeler, H. J., and B. L. Hartwell, paper on use of ammonium nitrate 53
work on association potash samples 60
Whitney, Milton, on sampling soils 108
Wiechmann's method for sugars 137, 150
Willard, J. T., work on association cattle-food samples 131
Williams, W. J., work on association samples 31, 60
Willits, Hon. Edwin, address of 98
Wine. (*See* Fermented Liquors.)
Winton, A. L., determination of water in cheese 13, 14
work on cheese analysis 13–15
Woll, F. L., method of standardizing acid 75, 76
work on association cattle-food samples 119, 124, 125, 129–131
Wood ashes, provisional methods for 231

Z.

Zimmermann, Dr., on association potash method 64